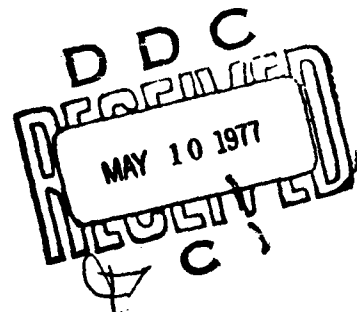ℝ𝕃

PROPERTIES RESEARCH LABORATORY

SPECTRAL AND TOTAL EMISSIVITY AND REFLECTIVITY
AT HIGH TEMPERATURES

1 Feb 76-31 Jan 77

FINAL

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March 31, 1977

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R.E. Taylor and D.P. DeWitt

Properties Research Laboratory

School of Mechanical Engineering

Purdue University, West Lafayette, Indiana

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Abstract

The automated high temperature emissometer for the accurate measurement of spectral emissivity from 0.6 to 11 micrometers and also total emissivity is described. This emissometer is used in conjunction with the multiproperty apparatus which measures a number of thermophysical properties on the same specimen. Spectral emissivity data for tantalum at 1500K measured with the emissometer is compared to literature values. These data were found to generally agree within 2% below 7 micrometers, but to diverge at longer wavelengths. With the use of improved amplifiers and of signal detectors with increased sensitivity, the accuracy above 7 micrometers is expected to improve significantly.

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INTRODUCTION

The Properties Research Laboratory (PRL) of the School of Mechanical Engineering, Purdue University, has developed a unique multiproperty apparatus for electrically-conducting solid materials at elevated temperatures. This apparatus is capable of state-of-the-art measurements on the same sample of the electrical resistivity, thermal expansion, and specific heat from room temperature to about 2600K and hemispherical total emittance and thermal conductivity from 1200 to 2600K.

The method basically involves Joulean heating of electrically-conducting rod or tubular samples supported between water-cooled electrodes and measuring voltage drops, temperature distribution, current flow and/or displacement of fiducial marks as the sample is heated. The experimental configuration can be readily adapted to spectral emissivity determinations at elevated temperatures and some preliminary data of rare earth oxides had been obtained at the start of the present program. The purpose of the present effort is to further extend the capabilities of the multiproperty apparatus by developing and testing a high temperature emissometer capable of yielding accurate (>99%) spectral emissivity data (0.6 to 11 micrometers) and total emissivity data at temperatures above 1200K on both electrical conducting and non-conducting materials.

The ability to measure such data at high temperatures is currently minimal. Literature searches have shown that insufficient data exists for sufficiently knowledgable predictions of material behavior in application of importance to current technology, especially in laser-related applications. Thus the development of this capability will result in a significant advance in our knowledge of thermal radiative properties.

PRINCIPLE OF THE METHOD

Spectral Emissivity Measurements

The spectral emissivity measurement technique is categorized as an integral blackbody method. The spectral emissivity is defined as the ratio of the spectral radiance from the sample surface to that from a blackbody both at the same temperature. By placing the blackbody in close proximity to the sample surface as shown in Figures 1 and 8, the temperature uniformity condition can be met. Since the radiance is viewed normally to the surface, the normal spectral emissivity is being measured.

The origin of the two quantities being sensed for the determination of the emissivity is shown in Figure 1, where $L_{\lambda 1}$ represents the radiance of the cavity which simulates a blackbody. The cavity is that of a cylinder of approximately 5.33 mm diameter with a small lateral hole of 1.32 mm diameter from which the spectral radiance emerges; the axial length of the cavity is nearly 29 cm when the nonconducting sample material is not mounted within the metallic cylinder and is 19 mm in length when such a sample is present.

From treatments by Siegel and Howell [1, see Page 260]* and by DeVos [2] the emissivity of the cavity—frequently referred to as the quality—can be estimated. $L_{\lambda 2}$ represents the spectral radiance from the sample which is the surface adjacent to the cavity opening or the surface as viewed through the upper lateral opening in the tube.

With the sample in plug form located directly behind the upper lateral hole within the same tube that is being used to simulate the blackbody cavity, the sample can be maintained at nearly the same temperature as the blackbody cavity, and the apparatus can be used to measure the emissivity of nonconducting materials. The functions of the metallic tube are then to provide the blackbody

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*Numbers in brackets refer to references listed at end of report.

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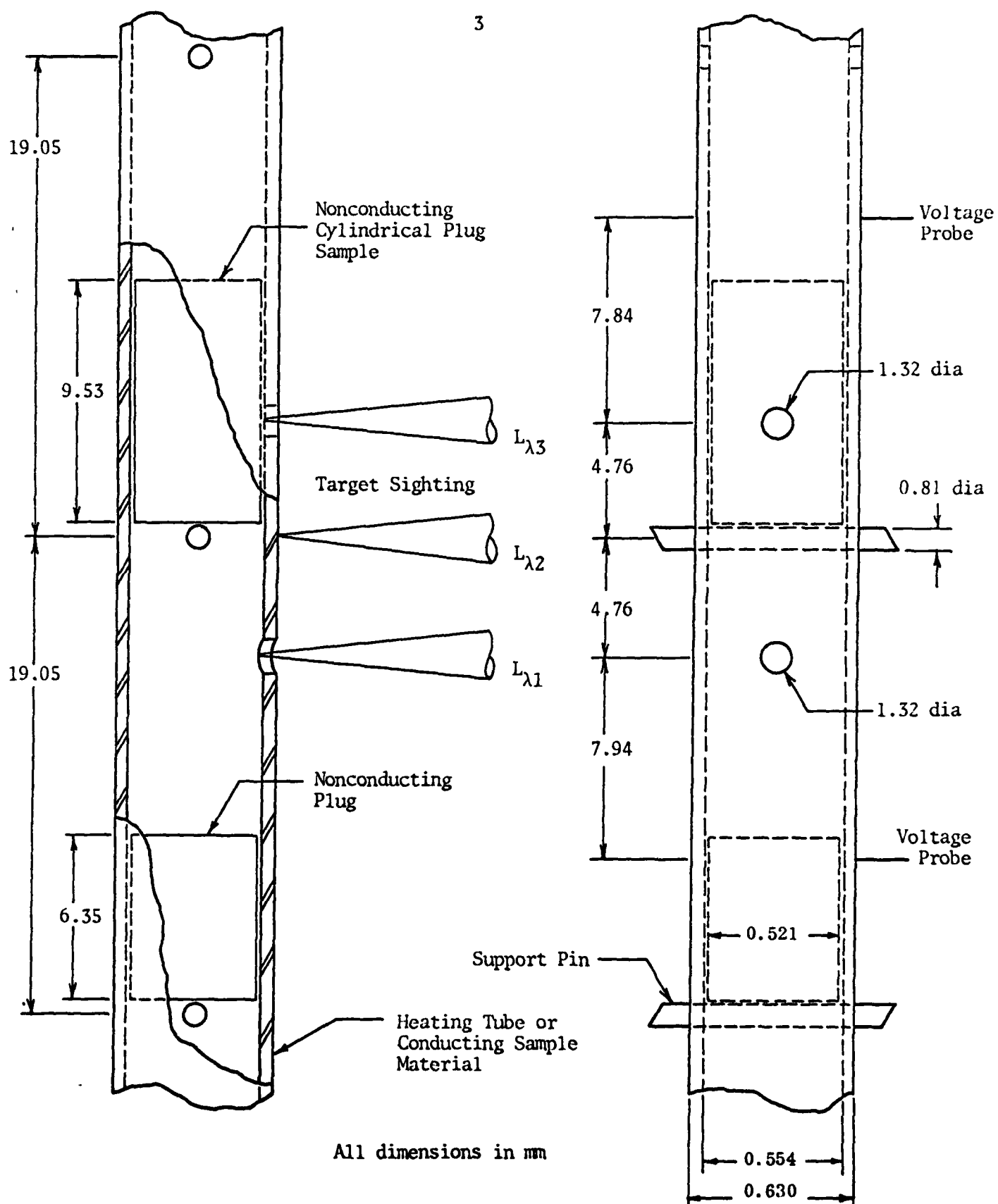
Figure 1. Target detail for the integral blackbody method for emissivity measurement

cavity and to heat the sample to a uniform temperature.

The emissivity is obtained from the ratio of two radiometric quantities. As a matter of experimental convenience, it is only necessary to obtain a detector signal that is proportional to the spectral radiance. In this procedure it is essential that the signal level for the zero radiance condition be known. This condition is referred to as the "radiometric zero" and hence the spectral emissivity is related to these observations as

$$\varepsilon_\lambda = \frac{S-Z}{H-Z} \tag{1}$$

where S = the signal proportional to the spectral radiance from the sample,

H = the signal proportional to the blackbody radiance,

Z = the signal obtained from a cold reference blackbody.

A description of the separate cold blackbody used to obtain the radiometric zero is subsequently presented.

Total Emissivity Measurements

The total emissivity, ε_t, is obtained by integration of the spectral emissivity over all wavelengths where the weighting function is the spectral radiance, $L_{b\lambda}(T)$, as prescribed by Planck's Law; that is

$$\varepsilon_t = \frac{\int_0^\infty \varepsilon_\lambda L_{b\lambda}(T)d\lambda}{\int_0^\infty L_{b\lambda}(T)d\lambda} \tag{2}$$

where ε_λ is the measured spectral emissivity. Notice that the denominator has the form of the well known Stefan-Boltzmann relation σT^4 where $\sigma = 5.669 \times 10^{-8}$ $Wm^{-2}K^{-4}$ and T has units of kelvins.

An alternate direct method of obtaining the total emissivity is to use a detector with a flat spectral response over the wavelength region where there is significant spectral radiance. These wavelength regions for various temperatures are illustrated in Figure 2 [Ref. 1] and are listed in Table 1, where x%
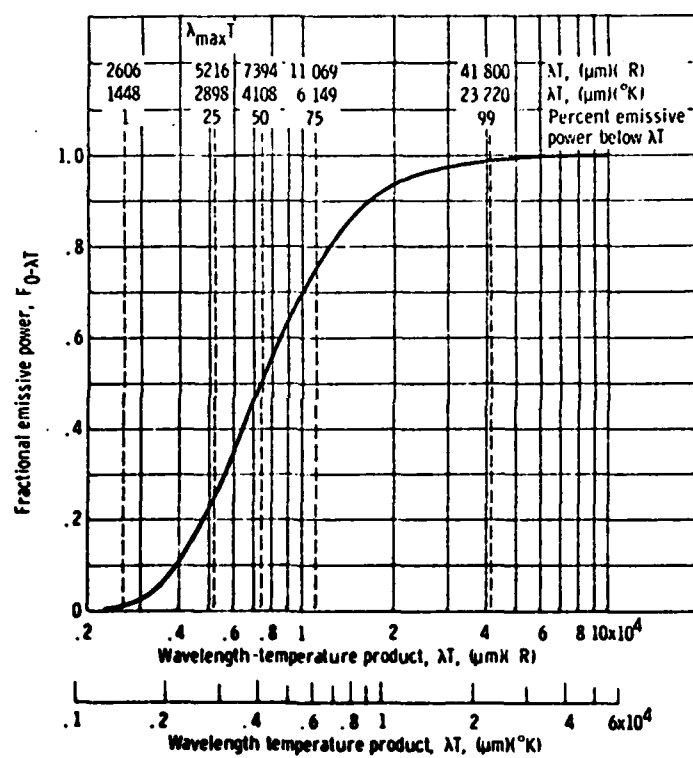
Figure 2. Fractional blackbody spectral radiance in the spectral region
0 to λT [Ref. 1]

Table 1

Wavelength Limits for Total Emissivity Calculation from Spectral Emissivity for Various Temperatures

T(K)	$\lambda(\mu m)$ limits - x%*			
	.5%	1.5%	98.5%	99.5%
1000	1.32	1.53	19.7	29.4
1500	0.88	1.02	13.1	19.6
2000	0.66	0.77	9.86	14.7
2500	0.53	0.61	7.89	11.7

*x% denotes the percentage of blackbody spectral radiant exitance in the wavelength region 0 to λ compared to the total exitance.

means the percentage of blackbody spectral exitance in the spectral range 0 to λ as compared to the total exitance.

The total emissivity of a sample may be found by the indirect method when Eq. (1) is substituted into Eq. (2) where S, H and Z are evaluated as follows:

$$S = \int_0^\infty \epsilon_\lambda L_{b\lambda}(T_t) A_t \omega_t R(\lambda) xGx\tau_{0t}(\lambda)d\lambda \qquad (3)$$

$$H = \int_0^\infty L_{b\lambda}(T_t) H_t \omega_t R(\lambda) Gx\tau_{0t}(\lambda)d\lambda \qquad (4)$$

$$Z = \int_0^\infty L_{b\lambda}(T_0) A_t \omega_t R(\lambda) xGx\tau_{0t}(\lambda)d\lambda \qquad (5)$$

where

$L_{b\lambda}(T)$ = spectral radiance of the blackbody at temperature T, $Wm^{-2}\mu m^{-1}sr^{-1}$

A_t = viewing area of the target (blackbody or sample) surface, m^2

ω_t = solid angle of the optical train in viewing the target, sr

$R(\lambda)$ = spectral responsivity of the detector, V/watts

G = gain setting of the amplifier

τ_{0t} = spectral transmittance of the optical train

T_t = temperature of the target, sample or blackbody

T_0 = temperature of the cold or radiometric zero blackbody

Later in our program, the effect of these parameters to cause systematic errors on total emissivity will be analyzed in detail.

Blackbody Considerations

A blackbody is defined as a surface that absorbs all of the radiant flux incident upon it and emits the maximum possible amount of thermal radiant flux or spectral radiance. Cavities of proper geometric shape and surface conditions

can be constructed which very closely approximate the performance of the ideal blackbody. In the Emissometer two such cavities are required to obtain emissivity data. One cavity, integral with the sample, is maintained at the temperature of the sample, 1000 to 2500 K, while the second cavity, located separate from the sample, is maintained at a much lower temperature, around 300 K. Recalling that the total exitance from a blackbody is proportional to T^4, it is readily seen that the radiant flux of a surface between 1000 and 2500 K is 2 to 4 orders of magnitude greater than a body at 300 K.

In order to achieve high quality blackbodies, the geometries of the cavities must satisfy certain criteria. For the case of the integral sample-blackbody the important parameters are the lateral hole opening radius r, the inner tube diameter D, and the axial half-length of the tube L. For our conditions, r/D = 0.12 and L/D = 26, and then assuming the inner surface of the tantalum is specular with an emissivity of 0.5, the DeVos theory predicts a quality of 0.993 as discussed in Reference 2. The effect of the quality being less than unity is to cause the measured emissivity to be systematically high by 1 - 0.993 or 0.7%. More detailed experiments are in progress to verify this result and the important influence it has on the emissivity measurements.

The shape of the radiometric zero reference or cold blackbody is cylindrical and the quality is determined by the depth x to diameter D ratio, x/D, and the emissivity of the surface. Based upon the Siegel and Howell model [1], for a diffuse emittance of 0.5 for graphite from which the blackbody is constructed and an x/D = 12.9, the quality of the blackbody is 0.999. The quality of this cavity is of little concern since the spectral exitance is extremely low as compared to that from the sample and since the spectral radiance resulting from any reflectance irradiance (from the sample) would likewise be low.

APPARATUS

Multi-Property Apparatus

This experimental apparatus has been described in detail previously [3, 4, 5, 6, 7] and only a brief description will be presented here. The apparatus is shown schematically in Figure 3.

The sample holder, in the form of a tube, is supported vertically between two electrodes. The upper electrode is movable in that it can be positioned at any height, but after positioning it is rigidly clamped. The lower electrode is movable and permits relatively free sample expansion and contraction. The electrodes are designed so that the specimen can extend axially through them. Thus, the electrode separation distance is adjustable and any portion of a long (up to 35 cm) sample may be positioned between the electrode clamps. To accomodate length changes due to temperature change, a strain relief system is provided with the lower electrode adjustable.

The sample holder is contained within a large vacuum bell jar which is raised and lowered by a power-driven hoist. Vacuum in the low 10^{-6} torr range is routinely achieved within a few hours and vacuum in the mid 10^{-7} torr range can be attained with longer pump-out times. Provisions to reduce backstreaming are incorporated into the vacuum system. The water-cooled bell jar features two vertical optical windows and an internal window shutter to protect one of the windows during the time intervals between measurements. The bell jar rests on a feed-through collar which contains rotary feed-throughs, instrumentation leads, electrical connections and water lines. Sensor signals--voltage probes, optical pyrometer, etc.--are patched into a terminal board connected to the minicomputer. Power to the sample and heaters is supplied by regulated DC power supplies. A 500 ampere-50 volt supply, transformer, calibrated shunt, reversing switches and remote control circuitry is used for sample operation at temperatures above 1800 K while three 100-amp Kepco Power Supplies are used for lower sample temperatures [8].

10.
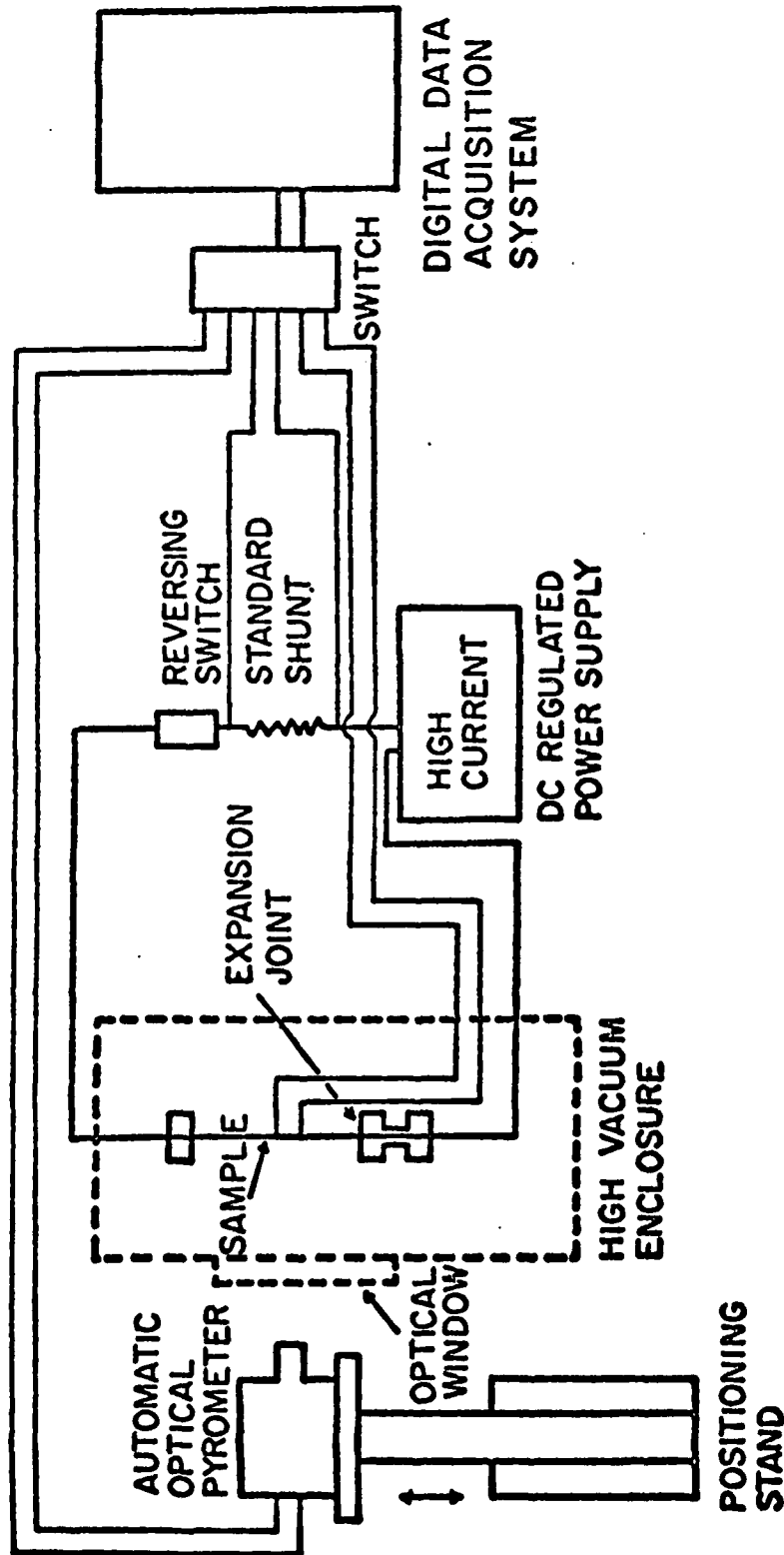
Figure 3. Schematic Diagram of Multiproperty Apparatus

MINICOMPUTER SYSTEM

General Description

The system contains a PDP-8/E minicomputer (central processing unit, CPU) with 24,000 words of core connected to an omnibus. Connected to the omnibus through a "data break" is a controller and disk drive for a disk system which is subdivided into four parts, System (SYS), disk 1, disk 2, and disk 3. The SYS system with 1×10^6 word capacity and DSK1 with 6×10^5 word capacity reside on a removable cartridge with floating head. The other two parts (DSK2 and DSK3 with 1×10^6 and 6×10^5 word capacity, respectively) reside on a fixed head cartridge. Information can be swapped readily among the various subdivisions. The capacity of the DDAS is sufficiently large (3.3×10^6 words) that programs and experimental data may be stored and used without input/output to any other device such as magnetic tape, punched tape, punched cards, etc., although a high speed paper tape reader/punch and magnetic tape units are included. Other disk cartridges may be readily substituted for the removable cartridge, providing additional storage and back up capabilities. Instructions and programs may also be entered through the Tektronix display terminal (No. 4010-1), which is the master input/output device or through the Decwriter (high-speed teletype) or standard teletype units. The Decwriter is essentially used as a line printer to give printed output. In addition, hard copies of whatever is displayed on the Display Terminal can be made by the Tektronix Hard Copy Unit (No. 4610).

Experimental data are directly entered into the system through an analog-to-digital converter (ADC) or through a VIDAR Model 521-01 integrating digital voltmeter (IDVM). The IDVM is controlled via a master Scanner (Vidar 610) and is equipped for 100 input channels. The Vidar DVM has full scale ranges from 10 MV to 1000 V in steps of 10 and has three integration periods (166.7, 16.7

and 1.67 milliseconds) so chosen as to essentially provide infinite rejection of 60 hz signals. The resolution of the IDVM depends upon the integration period and for the 10 MV full scale range is ±0.1 microvolt for the longest integration period. Operating in conjunction with the IDVM is a special crystal-controlled timer which accurately records the time the data are taken, without resort to software timing.

In addition, the DDAS is equipped with a real time clock and with programmable switches and digital-to-analog converters (DAC) for controlling experiments.

The DDAS is pictured in Figure 4. The first cabinet on the right of the four cabinets contains the disk and controller and also storage for magnetic tapes. The second cabinet from the right contains the minicomputer, magnetic tape drive and paper tape unit. The third cabinet from the right contains the integrating digital voltmeter and scanner. The cabinet on the left contains the floating point processor. Also visible in the foreground of Figure 4 is the Decwriter hard copy unit and visual display [9].

13.

Figure 4. Digital data acquisition system
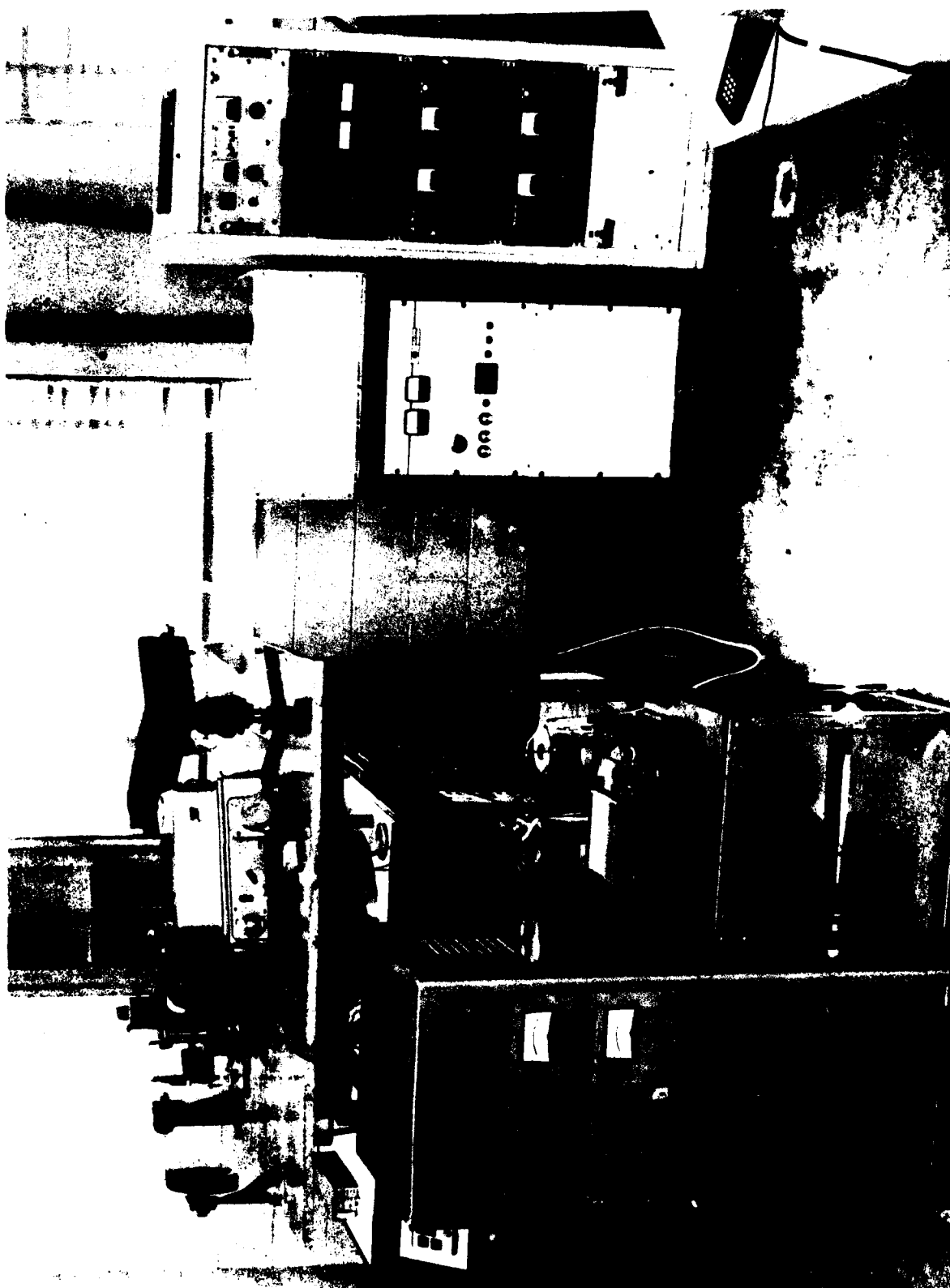
Figure 5. Emissometer showing DC power supplies on the right, amplifiers in front and the Multi-Property Apparatus in the background

DESCRIPTION OF THE EMISSOMETER

General Features

The Emissometer is mounted on an optical table located in front of the Multi-Property Apparatus. A general view of the Emissometer, Multi-Property Apparatus and power supplies is shown in Figure 5. Figure 6 is a scale drawing showing the placement of the various components of the optical transfer system, while Figure 7 is the optical diagram showing only the optical path of the target flux being received by the two detectors for the determination of either the spectral or total emissivity. Table 2 identifies the components of the Emissometer in greater detail.

Sample Configuration

Further modifications to the Multi-Property Apparatus include the replacement of the main viewing window of high quality Pyrex when using the radiation thermometer such as the Pyro optical pyrometer, with a potassium chloride window for use in the spectral region out to 15μm. This window may be seen in Figure 5 as the left viewing port. The other major modification was the inclusion of the water cooled radiometric zero reference blackbody. A detailed sketch of this addition is shown in Figure 8 indicating its location with respect to the sample holder.

Control Features to Emissometer

The minicomputer monitors and controls the acquisition of data as shown in Figure 9. The abbreviations on this schematic correspond to the items listed in Table 2.

In brief, the computer controls the following operations of the Emissometer: positions M1 such that the target viewed by the optical system is blackbody cavity, sample or radiometric zero blackbody; positions M2 such that the flux from the target is transferred to the monochromator MC (spectral emissivity

determination) or to the total radiation detector D-2 (total emissivity measurements); increments the wavelength drum position by a specified value. The computer monitors the detector signal strength and indicates, by a message on the display unit, that the operator must take corrective action. The computer records the signal levels from the detectors, D1 and D2, and from the pyrometer.

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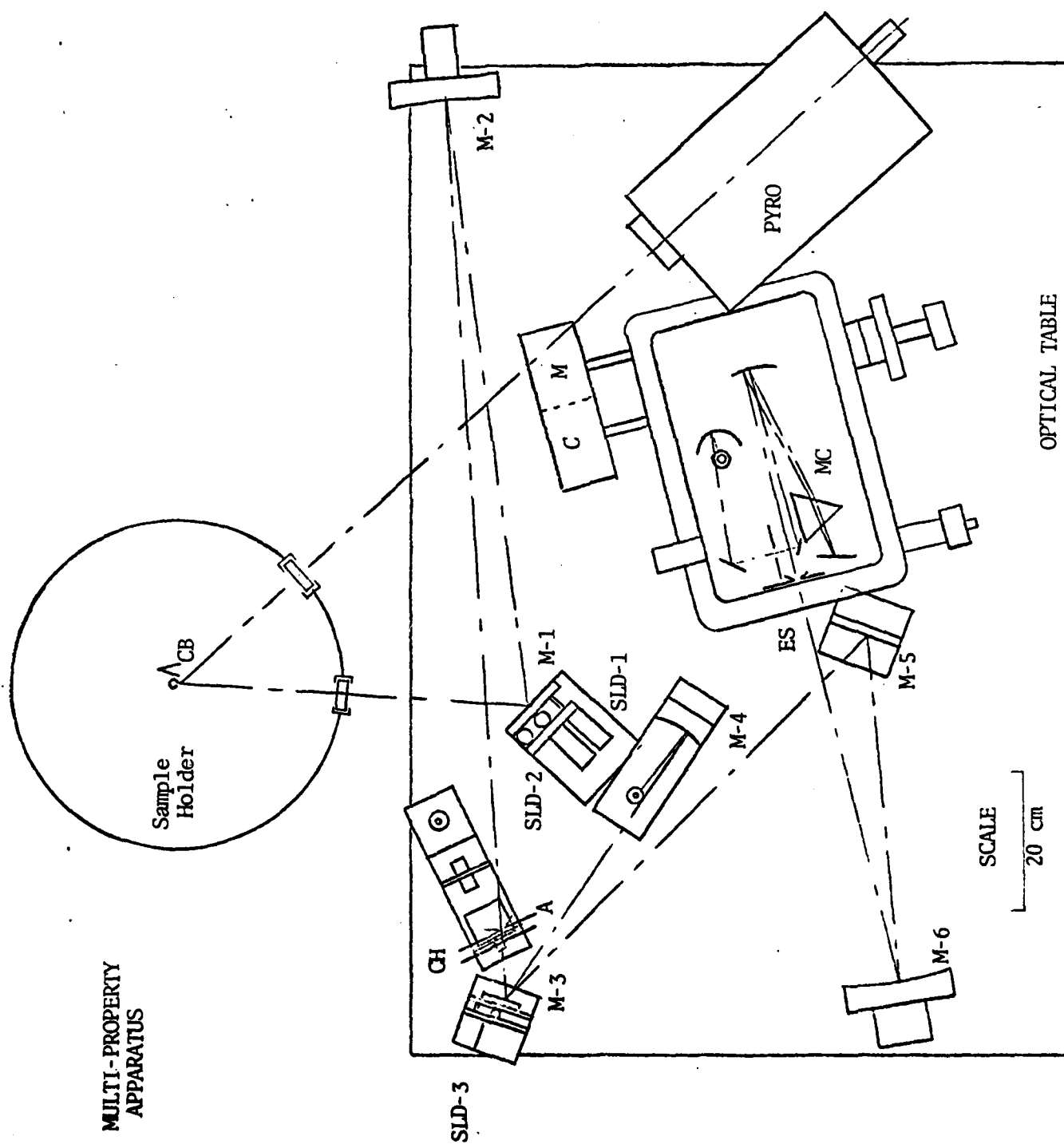
Figure 6. Schematic of the Emissometer illustrating positioning of sample, optical train, and detection system.

SPECIMEN

CB

W

M-2

SCALE
20 cm

M-1

C

A

M-3

M-3

D

M-4

ES

M-5

M-6

MIRROR	SIZE (CM)	FOCAL LENGTH (CM)
M-1	10 x 10	--
M-2	15.2 dia.	91.4
M-3	5 x 5	--
M-4	7.6 dia.	ellipsoidal
M-5	13 x 10	--
M-6	15.2 dia.	45.7

SYMBOL	DESCRIPTION
W	NaCl Window, 6 mm thickness
A	Aperture, 0.8 mm diameter
CH	Chopper, 13 hz (4 blades)
D	Thermocouple Detector
ES	Entrance Slit to Monochromator
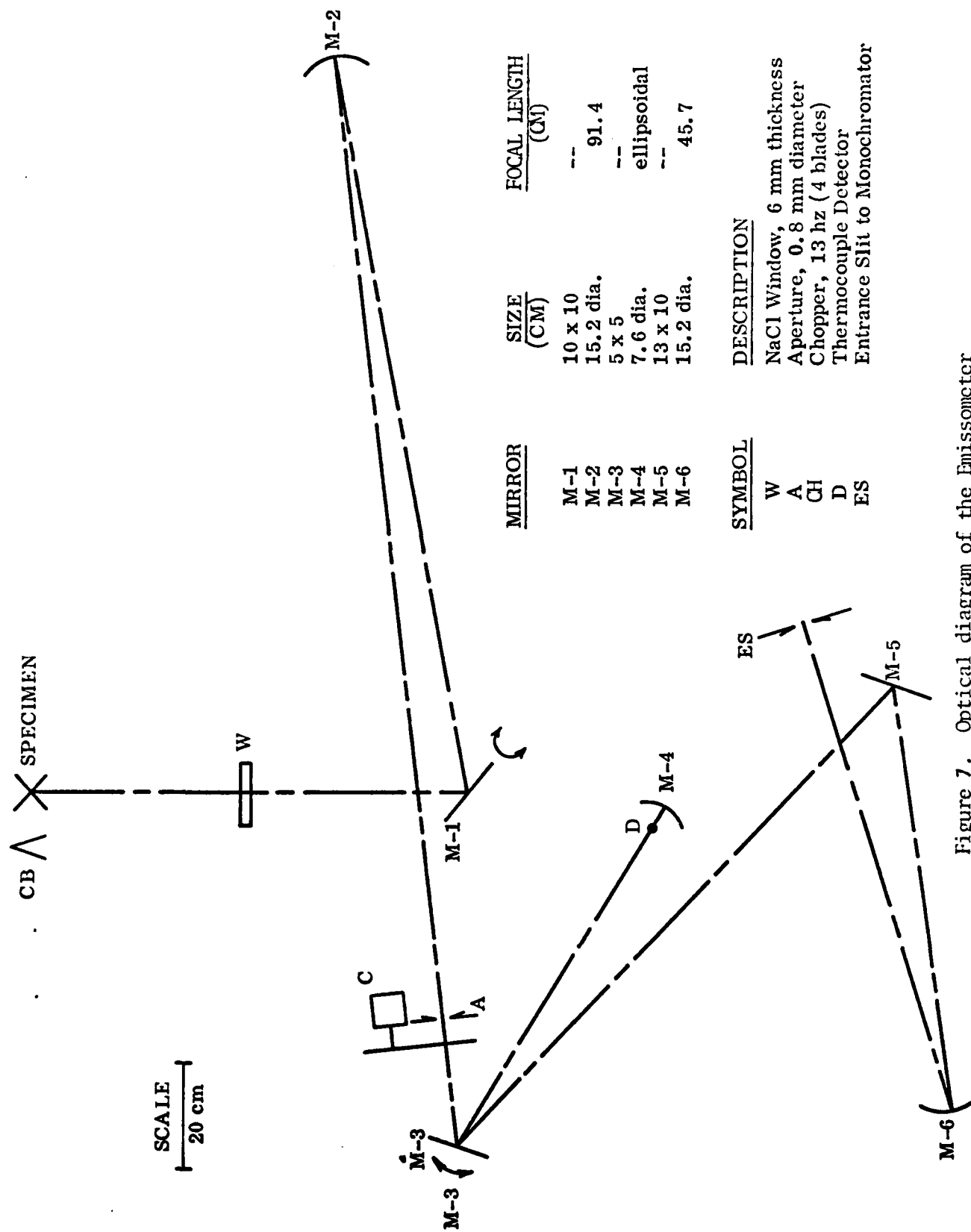
Figure 7. Optical diagram of the Emissometer

19.

Table 2

Description of Emissometer Components Represented in
Figures 6, 7, or 9

AMP-1   - Perkin Elmer Model 107 amplifier

AMP-2   - Princeton Applied Research Model JB-5 lock-in amplifier.
Monitored by computer switch number 42

C   - clutch for drive motor on monochromator

CH   - chopper or modulator

D-1   - detector mounted within the monochromator, vacuum TC or PbS cell

D-2   - spectrally flat vacuum thermocouple detector, 0.2×2 mm with
barium fluoride window

DPDT   - a simple double pole-double throw switch for operation of a
solenoid controlled mirror, M-3, focused on an optional
detector, D-2 (not shown). Controlled by computer switch #128

M   - the drive motor for the monochromator which is engaged and
disengaged by C and turned on and off by the computer under
switch number 64.

M-1   - a flat, 10×10 cm (4"×4") mirror directionally controlled by SLD-1
and SLD-2. Controlled by computer switch #16 and/or #32.

M-2   - a 15.2 cm (6") diameter concave mirror

M-3   - a flat 5.1×5.1 cm (2"×2") mirror directionally controlled by
SLD-3. Controlled by DPDT.

M-4   - a 7.6 cm (3") diameter ellipsoidal mirror

M-5   - a flat 12.7×10.2 cm (5"×4") mirror

M-6   - a 15.2 cm (6") diameter concave mirror

Table 2 Continued

MC   - a Perkin Elmer Model 98 Prism Monochromator with wavelength drive (M, C, POT) and detector D-1

P AMP   - P-E Thermocouple Preamplifier

POT   - a 200 Ω,20 turn spiralpot by Granine Controls Corp. Serial No. 544; monitored by computer terminal numbers 40 and 41

PYRO   - a Pyro Precision Pyrometer by Photomatic Pyrometer Instruments Co., Inc. monitored by computer terminal number 44

REF   - two 1.5 volt batteries as a reference for the potentiometer denoted POT

SLD-1   - an electric solenoid used to position M-1 such that the target is the radiometric zero blackbody or the lower (or upper) hole of the sample tube. Controlled by computer switch number 16

SLD-2   - an electric solenoid used to position M-1 such that the target is the upper or lower holes of the sample. Controlled by computer switch 32

SLD-3   - an electric solenoid used to position M-3 such that the target is imaged on the detector D-2 for total emissivity measurements or on the entrance slit of the monochromator, MC. Controlled by computer switch number 128 through DPDT

21.

19 ± 13

8.7 ± 0.8

Key:

S - Sample, Upper Lateral Hole
H - Blackbody Cavity
Z - Radiometric Zero Cavity

106.4

92.1

19.1

S

Z

H

6.40 dia

Radiometric Zero
Blackbody

6.30 OD×5.54 ID×292 Length Tube

All dimensions in mm
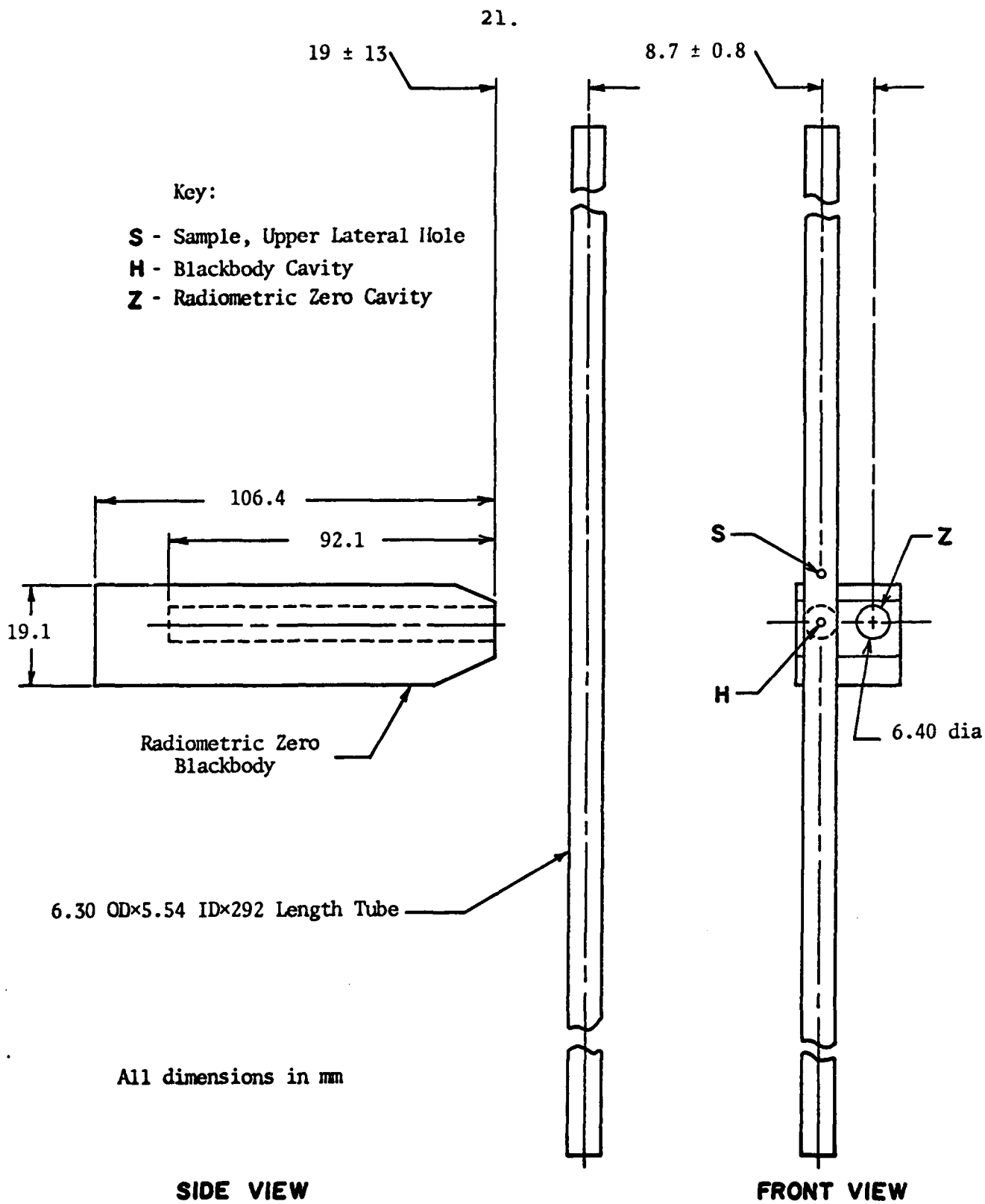
SIDE VIEW

FRONT VIEW

Figure 8. Location of the sample, blackbody cavity and radiometric zero
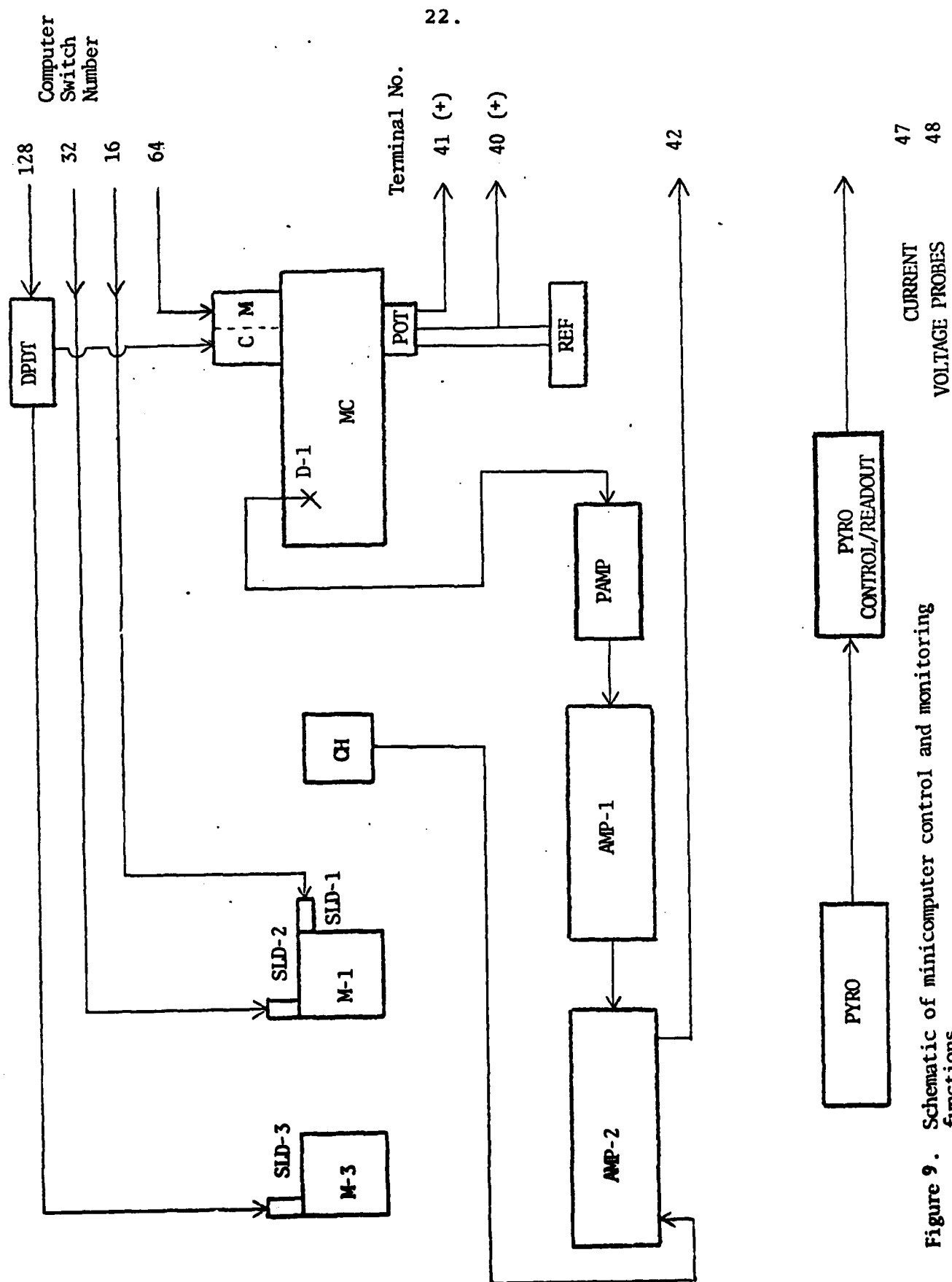targets

Figure 9. Schematic of minicomputer control and monitoring functions

PROCEDURE

If the sample material is a good conductor then it is formed into a thin walled cylinder with a small lateral hole bored at the midlength of the specimen to serve as a blackbody aperature. If the sample material is a non-conductor then a small solid cylindrical sample is placed in a cylindrical holder, usually tantalum, similar to the conducting sample described above except that two holes are bored near the center of the thin walled cylindrical tube. One hole serves as a blackbody aperature while the other hole serves as a viewing port for the non-conducting sample.

The cross sectional area and density of the cylindrical tube are determined. Voltage probe wires are spot-welded to the sample and the distance between the voltage probe wires is accurately determined.

The cylindrical tube is supported vertically between two adjustable electrodes within the bell jar; the lower electrode being connected to a strain relief mechanism which permits relatively free expansion and contraction of the tube. Voltage probe leads from the cylindrical tube are spot-welded to connecting leads within the bell jar. These leads pass from the bell jar through a multiple headed hermetic terminal, to the computer input terminals. A vacuum in the low 10^{-6} torr range can be achieved in the water-cooled bell jar in a relatively short time (in 15 minutes). A vacuum in the mid 10^{-7} torr range can be achieved with longer pump-out times. The sample is pre-treated by heating under high vacuum to a temperature higher than that for which measurements are desired.

The pyrometer, wavelength position potentiometer, and the mirror positioning solenoids are connected to computer input terminals. The potentiometer provides a signal which is proportional to the angular position of the spectrometer prism and voltage reference signal. Program SEM1 (Appendix A) takes the ratio formed by these two signals and compares it to predetermined ratios to determine

the wavelength of the radiation reaching the detector. The optics of the Emissometer are now aligned in such a way that the clearest image of the sample holder appears at the 0.8mm diameter aperture and is checked using a viewing apparatus designed specifically for this purpose. Mirrors M2 and M6 (Figures 6 and 7) are now finely adjusted such that the signal from the detector is maximized.

The spectral emissivity can be obtained by adjusting mirror M-1 (Figure 6) such that it looks at the sample, the blackbody and a zero reference. The program SEM 1 (Appendix A) controls these operations and plots the spectral emissivity as a function of wavelength. The configuration of the Emissometer permits generation of total emissivity data by repositioning of mirror M3 (Figure 6) causing the target radiance to reach the detector. The total emissivity along with current, voltage, temperature, and resistance can also be obtained through a second computer program. A useful check of the spectral emissivity can be obtained by integrating over the entire range of wavelengths and comparing the value to the total emissivity.

In the blackbody quality study the Emissometer was not used. The optical pyrometer is focused on one of three target areas - upper cavity, sample surface (lateral tube area located midway between the cavities), and lower cavity at three different temperatures. The configuration of Case I (Figure 9) is first observed and the radiance temperatures of the three target areas are recorded. Because this cavity configuration is nearly ideal, then the observed radiance temperature is the sample temperature, that is $T = T_{c,L} = T_{c,u}$. The spectral emissivity of the sample surface for Case I can be calculated from the observed spectral radiance temperature T_S and the sample temperature T. The spectral radiance temperature, T_S, is defined by the relation

$$L(\lambda, T_S) = \epsilon_\lambda L(\lambda, T) \qquad (6)$$

and using Wein's approximation to the Planck Distribution Law for spectral radiance from a blackbody, the emissivity can be related to the observed temperatures

$$\varepsilon_\lambda = \frac{\exp\ (-C_2/\lambda T_S)}{\exp\ (-C_2/\lambda T)} \tag{7}$$

Using this relation, the emissivity of the sample surface for Case I (tantalum) can be calculated.

The sample (tube) temperatures for the cavity configuration Cases II and III cannot be accurately determined by the optical pyrometer. The emissivities of all the cavities are less than unity; the emissivity of the tantulum tube does not remain sufficiently stable with repeated cycling that we could infer the temperature from the observed sample radiance temperature T_S and the spectral emissivity as determined from Case I measurements.

The procedure for determining the sample temperature T for Cases II and III based upon the resistance approach is as follows: Using the voltage probes located on the tube as shown in Figure 10, the potential drop across the test section is measured. The resistance of this section, R, is calculated from this potential drop (v) and the tube current, I, measured as described in Reference (10). For the observations on the three configurations at any one temperature level, the sample temperatures for Case II and Case III are determined by adjusting the tube current until the resistance of the test section is within a few percent of the value of Case I. Using the linear relationship between resistance and temperature, then

$$T_j = T_1 \frac{R_j}{R_1}\ \ j = 2,3 \tag{8}$$

where the subscripts refer to the cavity configuration case number.

With the sample temperatures for the configurations of Case II and III determined, the emissivity of the target areas can then be calculated from

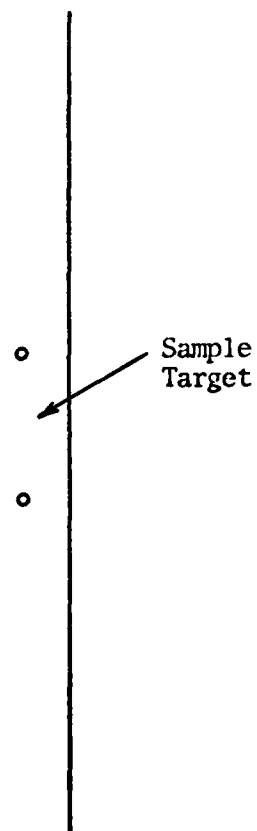26.
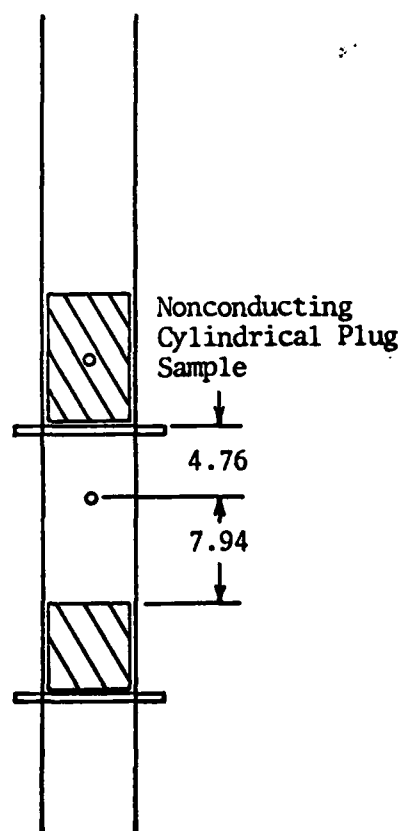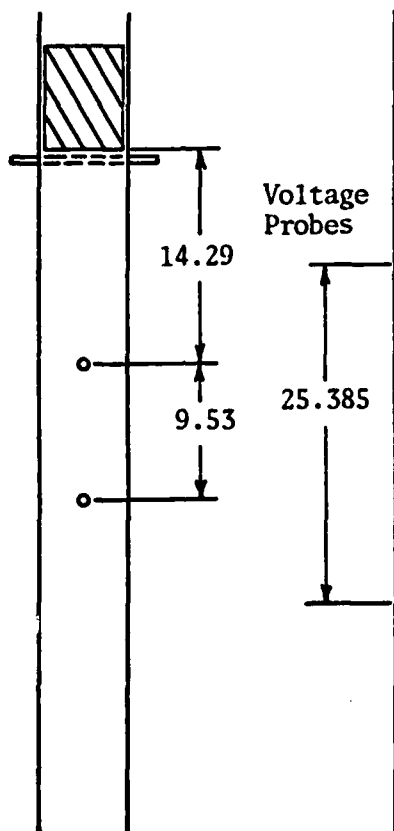
All dimensions in mm

Figure 10.   Configuration of blackbody cavities

Eq. 7. In using this relation, the radiance temperatures of the cavities $T_{C,L}$ and $T_{C,U}$ are used with the sample temperature T to calculate cavity emissivities.

RESULTS

Evaluating Performance Factors

The effect that various factors have on the spectral emissivity results
have been evaluated. All of the measurements subsequently discussed are for
the sample material tantalum. The effects to be reported upon include slit
masking, wavelength interval spacing, automatic vs manual scale changing, and
reproducibility of the emissivity spectra.

In an effort to increase the signal to noise ratio, masks were made to
partially cover the slits on the spectrometer and reduce any unwanted signal
from an outside source going into the spectrometer. In effect, the masks
reduce the slit height from 21 mm to 3.2 mm such that the target slightly
overfills the entrance aperture. As can be seen by comparison of Figure 11
without the mask and Figure 12 with the mask, there is no appreciable change
in the spectral emissivity. In these figures, the computer file reference
number, SEXXX, is shown in the upper left hand corner. This figure is a hard
copy of the display generated by the computer upon completion of the data
collection mode. The points represent the data observations, the upper set
corresponding to the blackbody (H) reading and the lower set (S) from the
sample. The abrupt changes in these points at 1 and 2.8μm correspond to ampli-
fier gain setting changes.

The effect on the emissivity spectra of the wavelength interval spacing
between data points is shown in Figures 13, 14 and 15. For these tests, fused
quartz is the prism material and the vacuum thermocouple is the detector.
Direct comparison of the three curves shows that the emissivity spectra have
generally the same shape but smoothness is more apparent when the wavelength
increment is largest. This is primarily a consequence of the manner in which
the computer generates the displayed curve, namely by straight line connection

29
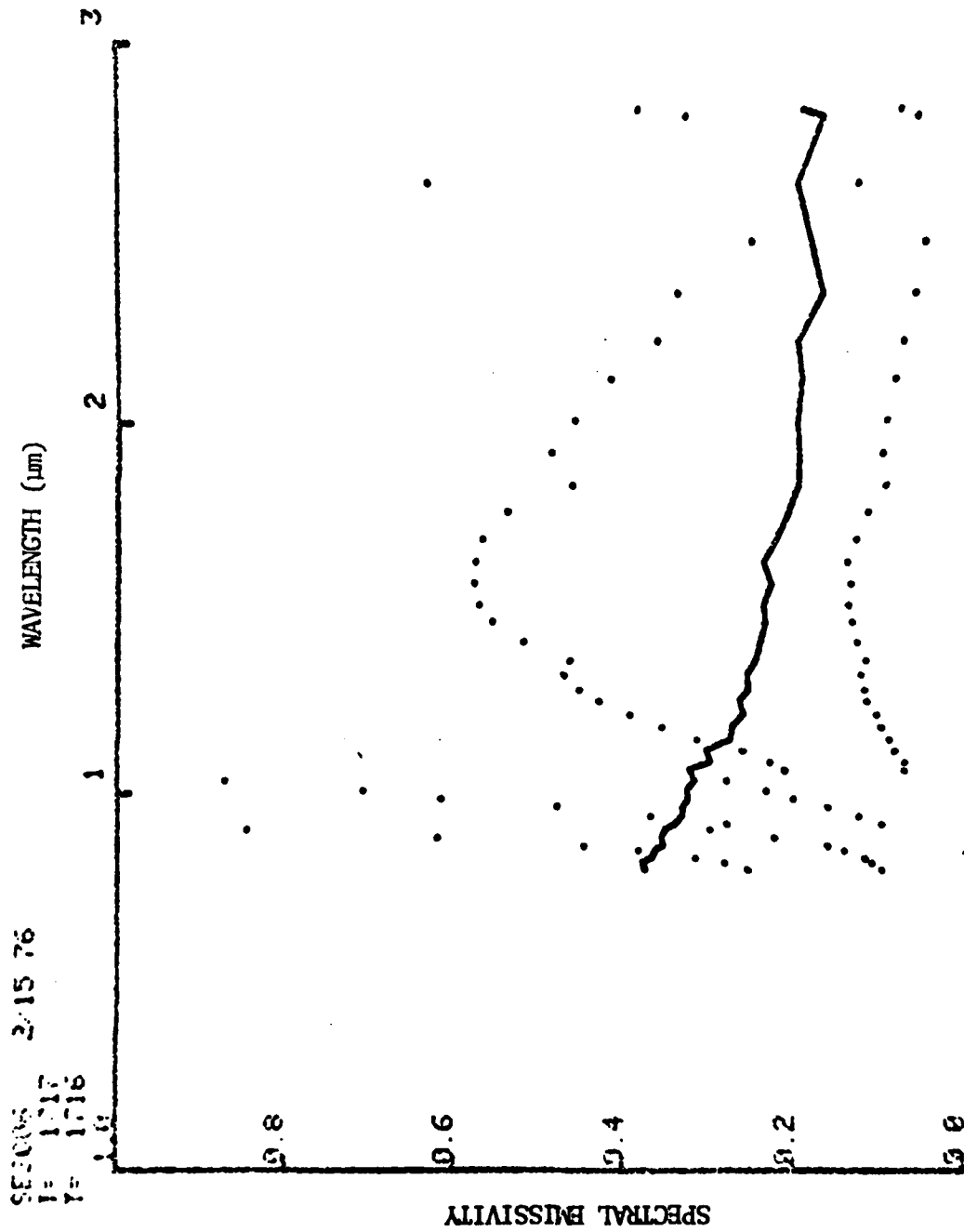
Figure 11. The effect of entrance slit masking on the emissivity spectra of tantalum (a) no mask on entrance slit
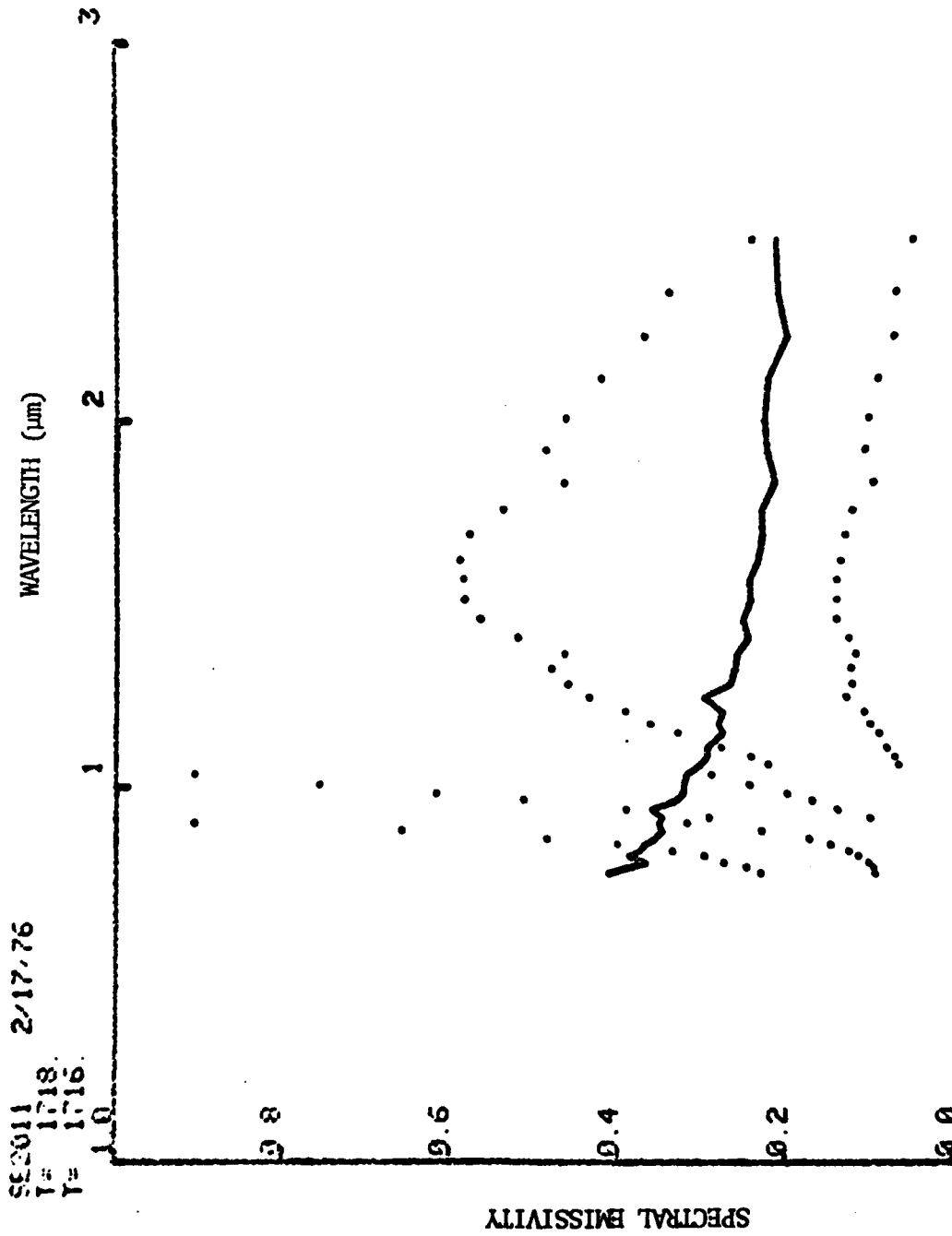
Figure 12.   The effect of entrance slit masking on the emissivity spectra of tantalum
(b) mask reduces entrance aperture and target slightly overfills aperture

WAVELENGTH (μm)

SPECTRAL EMISSIVITY
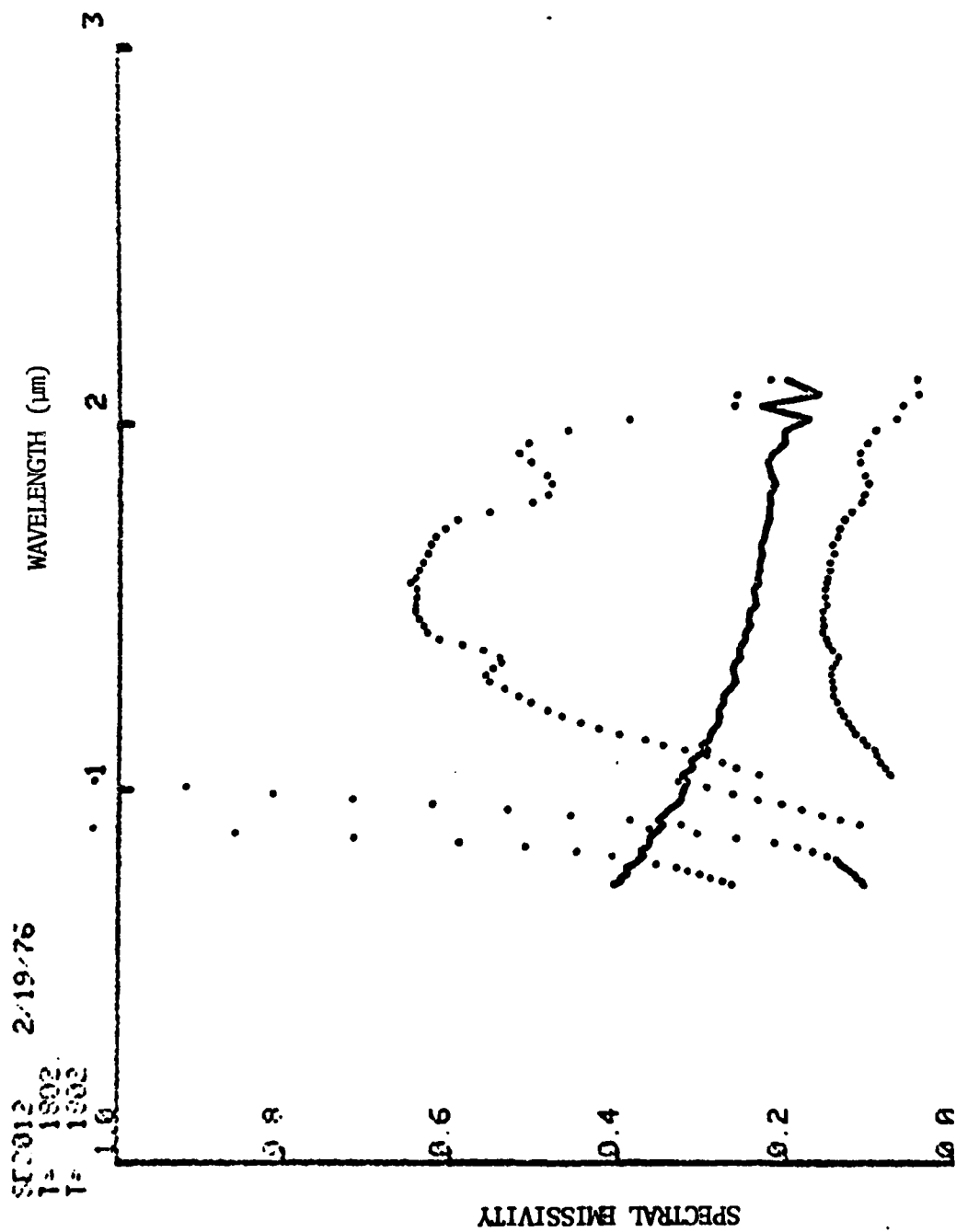
Figure 13. The effect of wavelength interval spacing on the emissivity spectra of tantalum, 1 to 2.8 μm (a) Δλ = 0.01 eV

32
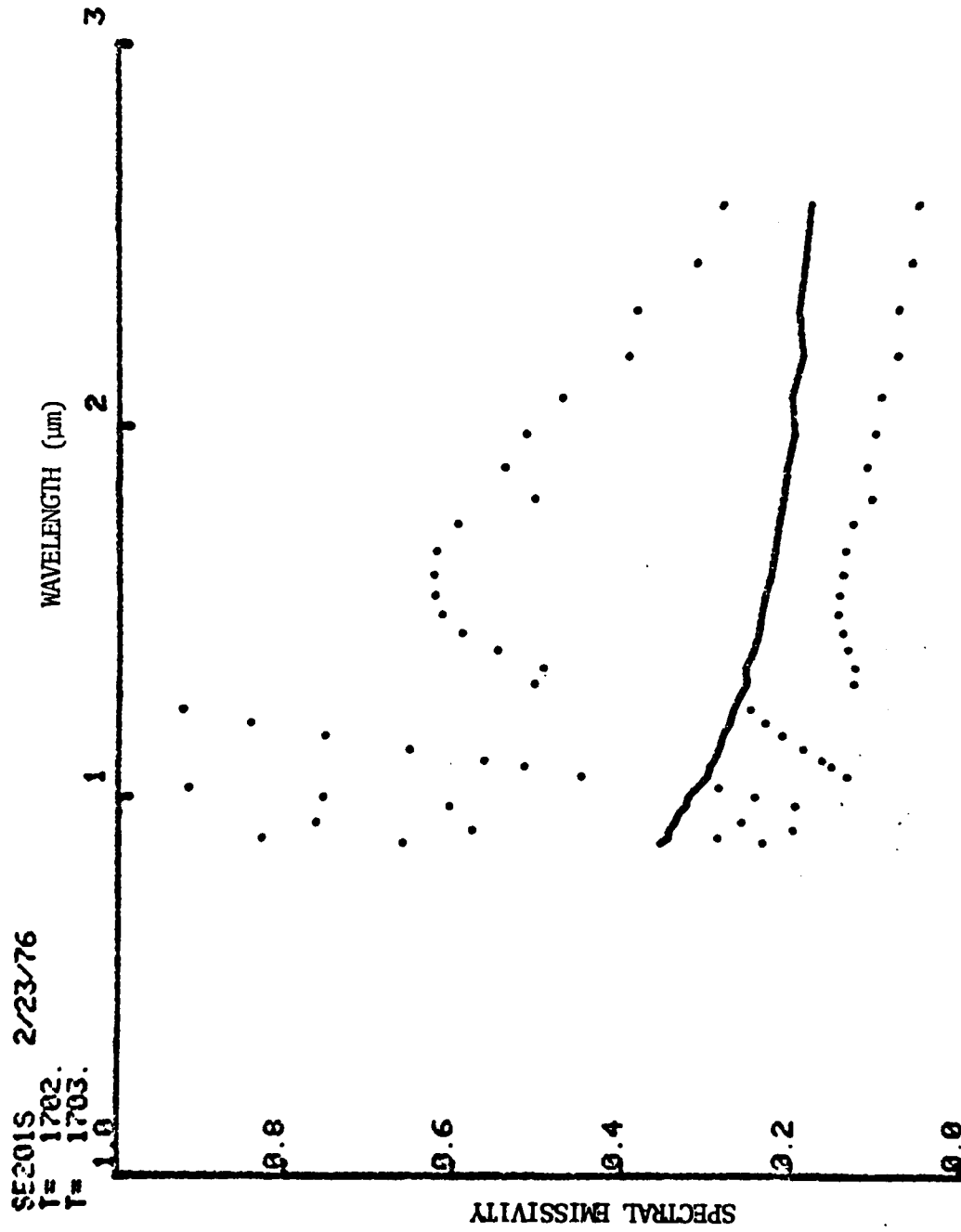
Figure 14. The effect of wavelength interval spacing on the emissivity spectra of tantalum, 1 to 2.8 μm (b) Δλ = 0.02 eV

33
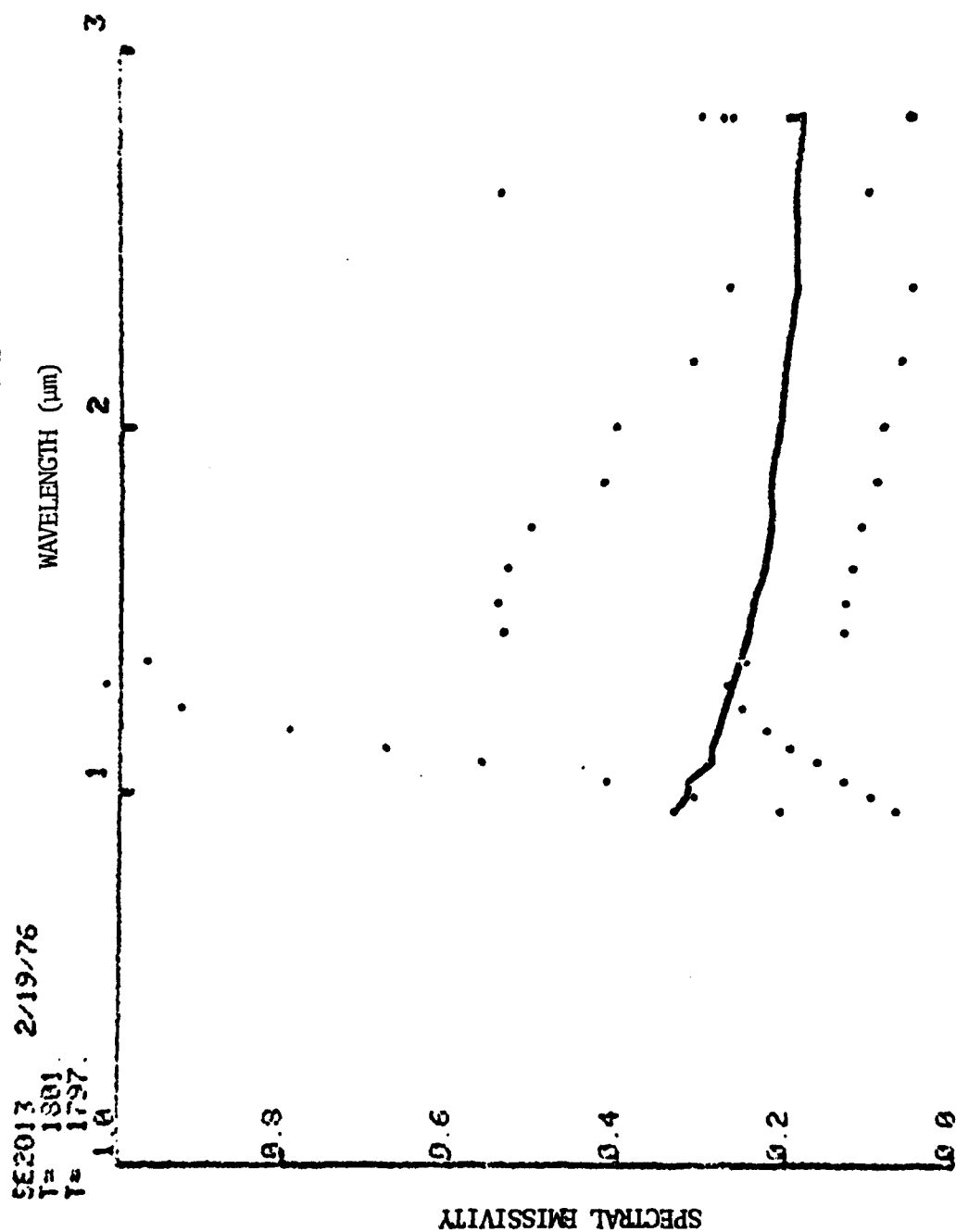
Figure 15. The effect of wavelength interval spacing on the emissivity spectra of tantalum, 1 to 2.8 μm (c) $\Delta\lambda = 0.05$ eV

between observed data points.

Data can be taken both automatically and manually. The next four graphs
(Runs SE2023, SE2049, SE2051, SE2056; Figures 16, 17, 18 and 19, respectively)
demonstrate the two methods of data acquisition. Figure 16 (SE2023) represents
an automatic run while Figure 17 (SE2049) represents a manual run, both at 1700
K. Here the automatic run appears to be slightly smoother than the manual
run. Figures 18 and 19 (SE2051 and SE2056) represent a manual run and automa-
tic run, respectively, at 1500 K. Observation of these two graphs would lead
one to believe the manual scale changes yielded better results here. Thus
from these two results, it is not clear whether automatic or manual gain changes
are likely to produce better results.

Once again data was taken for the purpose of showing the effect of the
spacing of data points in a run while using a salt prism in the spectrometer.
To illustrate the results, compare Figure 18 (SE2051) with Figures 20 and 21
(SE2058 and SE2057). The plotting done here, as in all past data runs, has been
produced by means of spacing the data points at equal increments of hundredths
of an eV or electron volt. The wavelength spacings for Figures 18, 20 and 21
(SE2051, SE2058 and SE2057) points are 0.03 eV and 0.01 eV, respectively. As
can be seen from these three figures, the results for the sodium chloride prism
and for the fused quartz prism show the same effect, namely the closer the data
points, the higher the frequency of variation from an averaged smooth curve.
It should be noted that all three of the curves or graphs were obtained with
manual changes in amplifier gain settings as described previously.

Curve Figure 22 (SE2024) has been added as an example of the reproducibil-
ity of the system. Figure 22 (SE2024) and Figure 21 (SE2057) represent measure-
ments taken under similar conditions more than six weeks apart. Upon examina-
tion of these two figures, one finds that this system is quite capable of
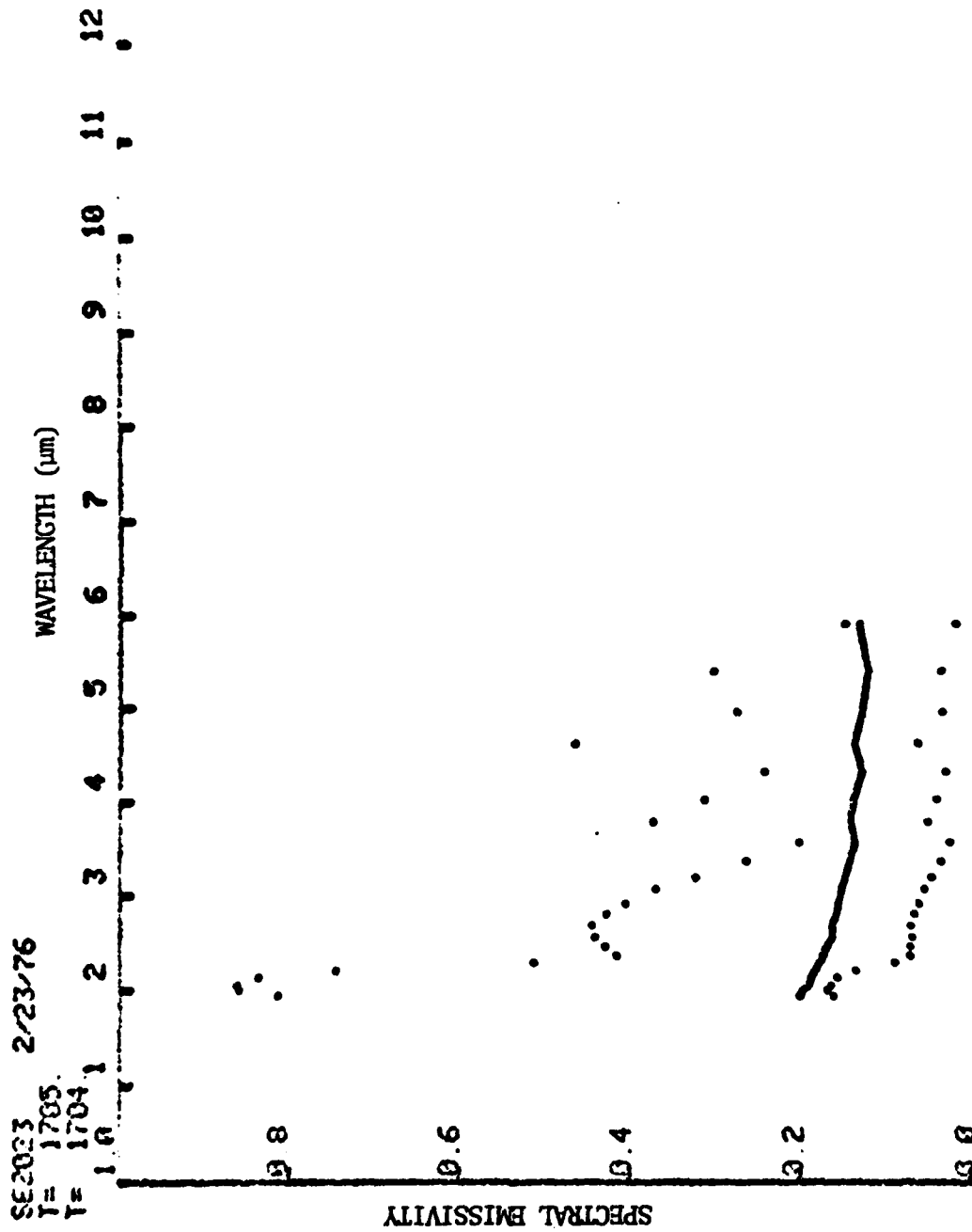
Figure 16. The effect of automatic vs manual scale changes at 1700 K on the emissivity spectra of tantalum, (a) 1.5 to 6.0 μm
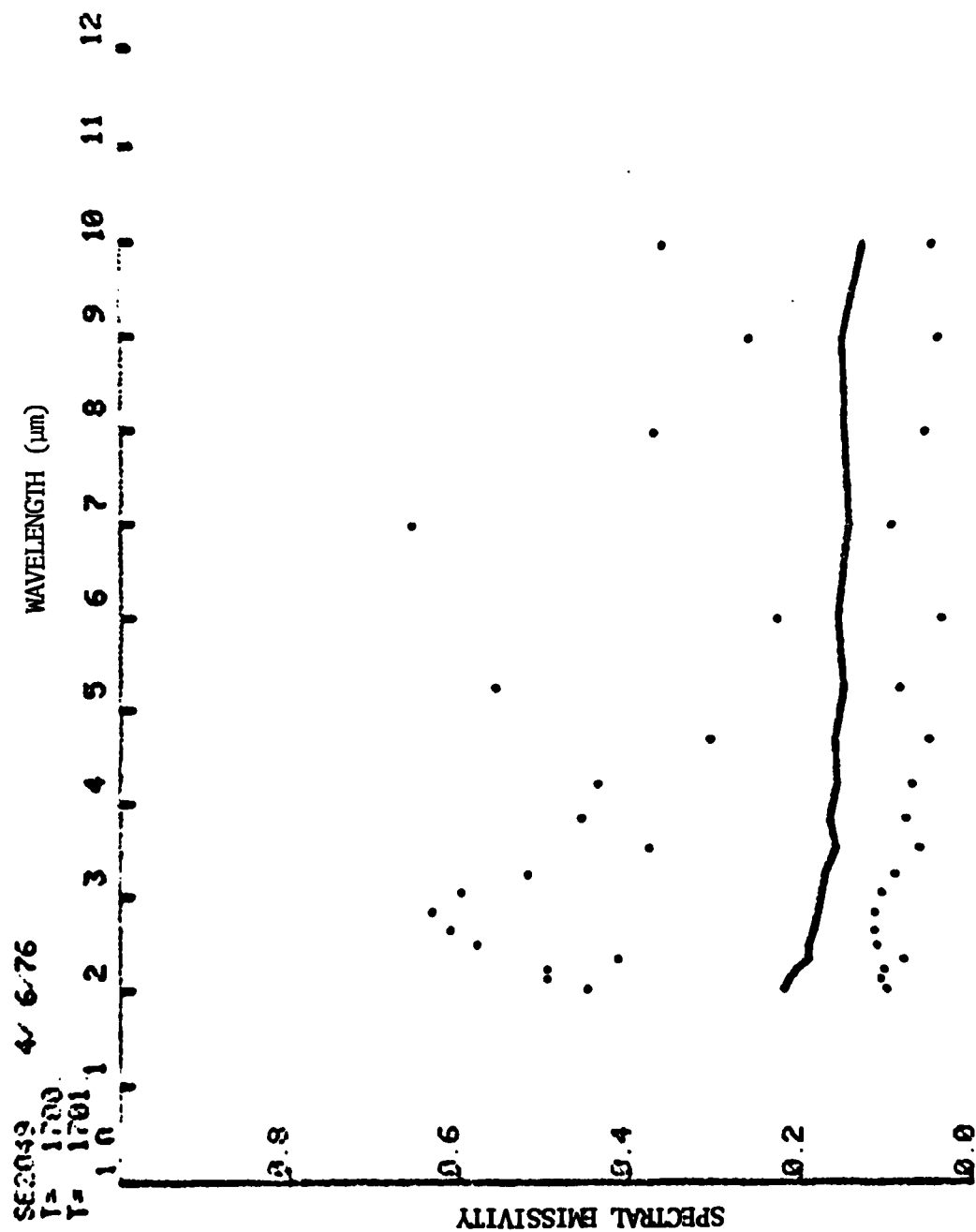
Figure 17. The effect of automatic vs manual scale changes at 1700 K on the emissivity spectra of tantalum, (b) 1.5 to 9.5 μm

WAVELENGTH (μm)

SPECTRAL EMISSIVITY

SE2051 4/ 6/76
T= 1501
T= 1503
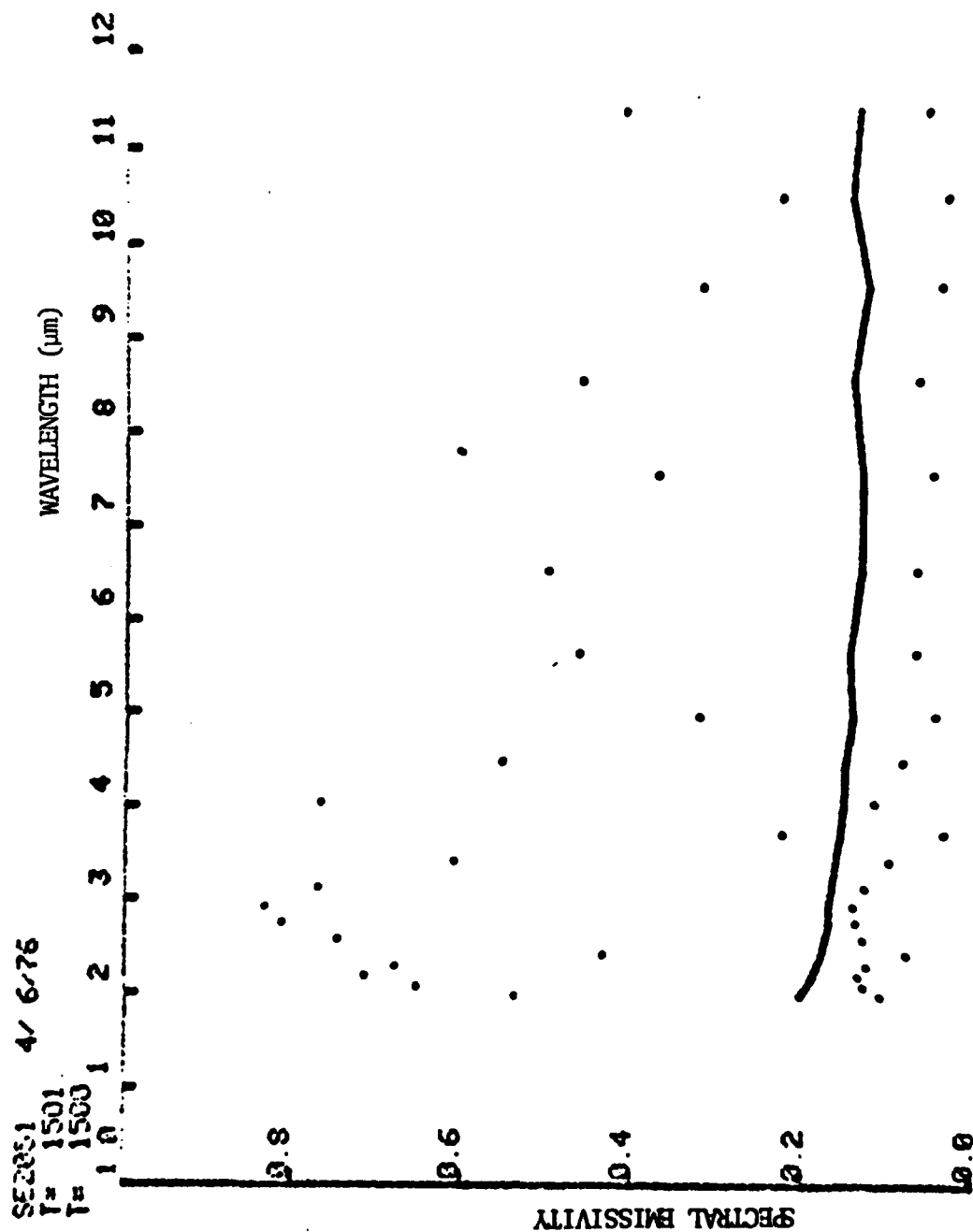
Figure 18. The effect of automatic vs manual scale changes at 1500 K on the emissivity spectra of tantalum, 2.0 to 11.5 μm, (a) Run SE2051

38
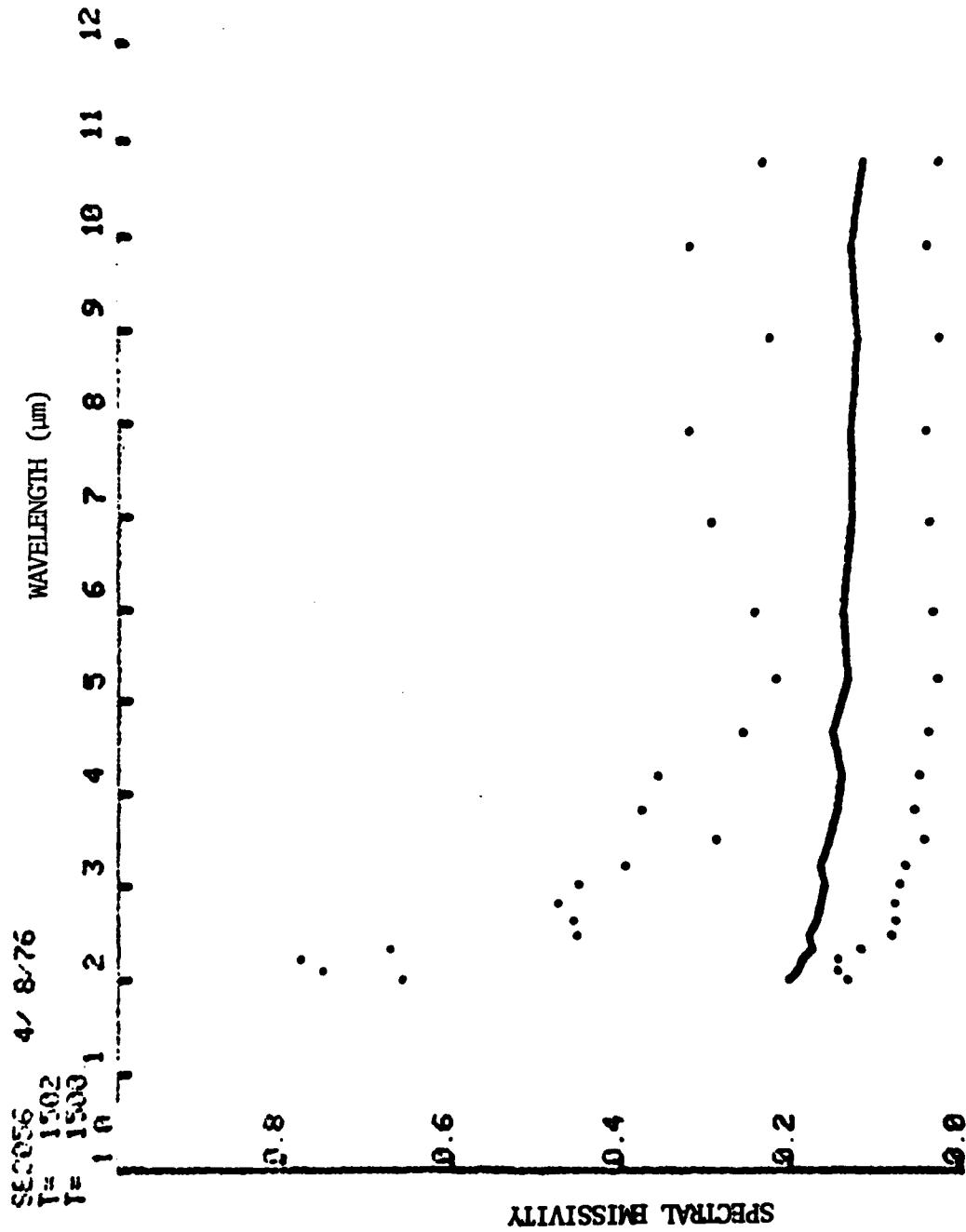
Figure 19. The effect of automatic vs manual scale changes at 1500 K on the emissivity spectra of tantalum, 2.0 to 11.5 μm, (b) Run SE2056

39.
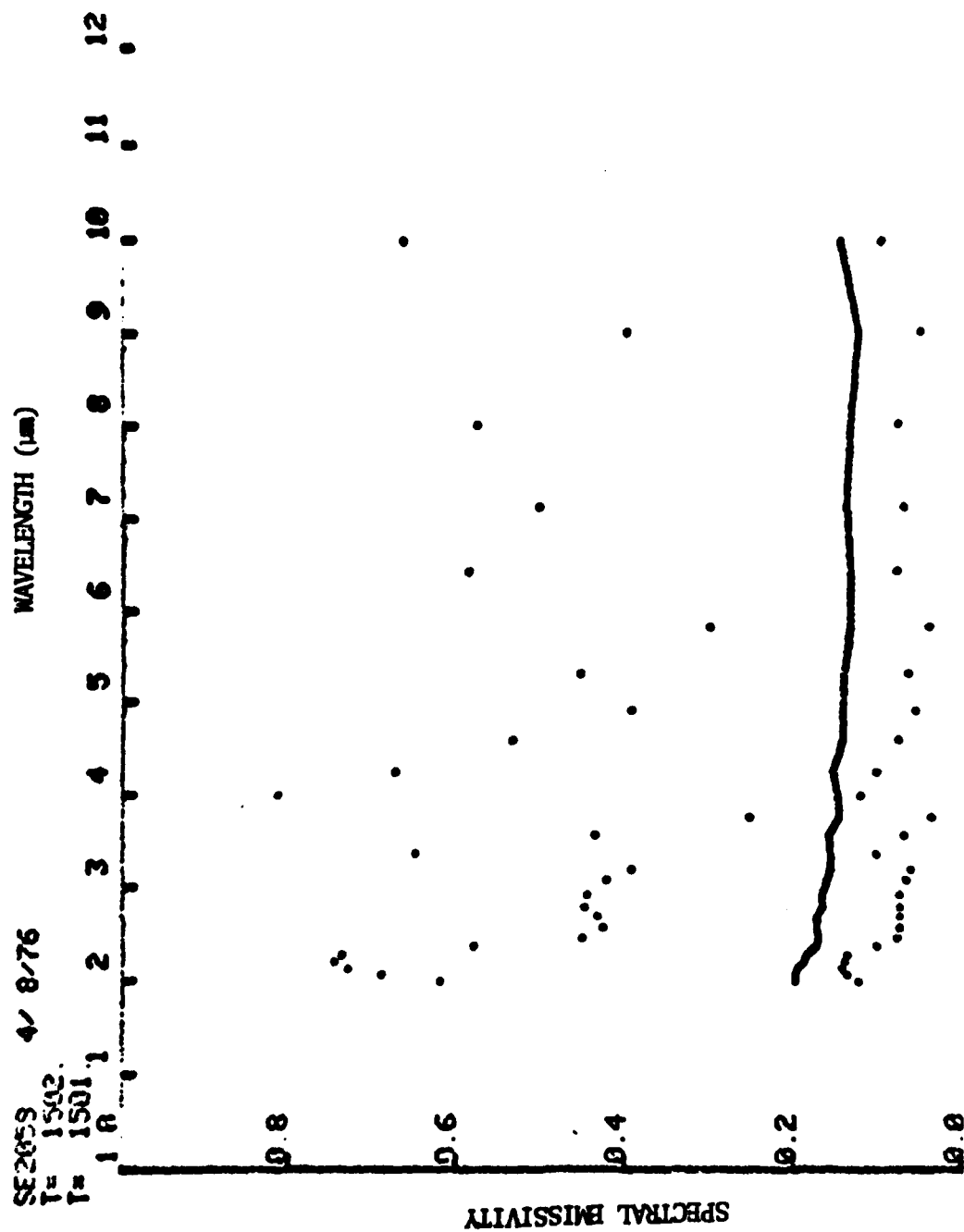
Figure 20. The effect of wavelength interval spacing on the emissivity spectra of tantalum, 2 to 10 µm (a) Δλ = 0.02 eV

40.
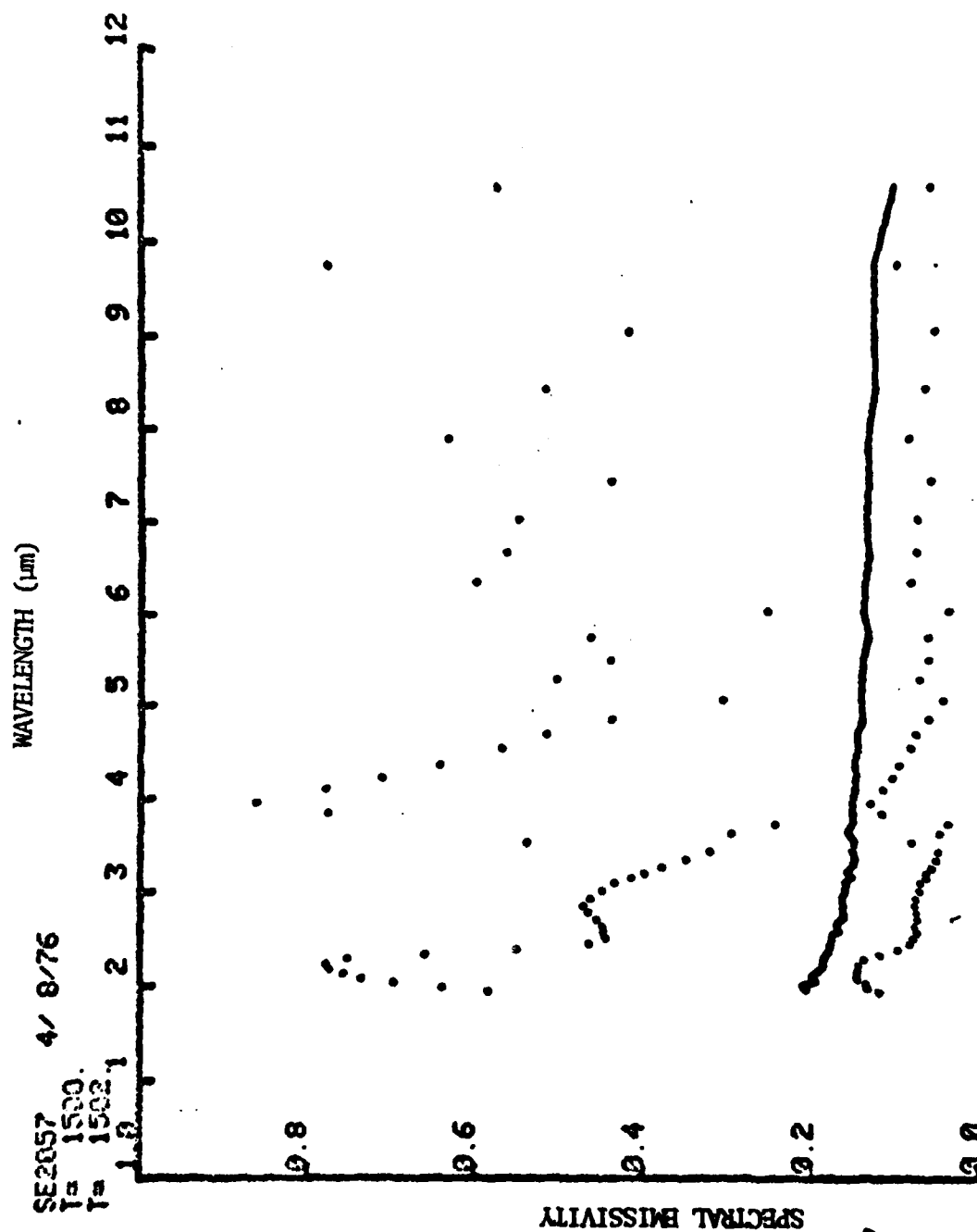
Figure 21. The effect of wavelength interval spacing on the emissivity spectral of tantalum, 2 to 10 µm (b) Δλ = 0.01 eV
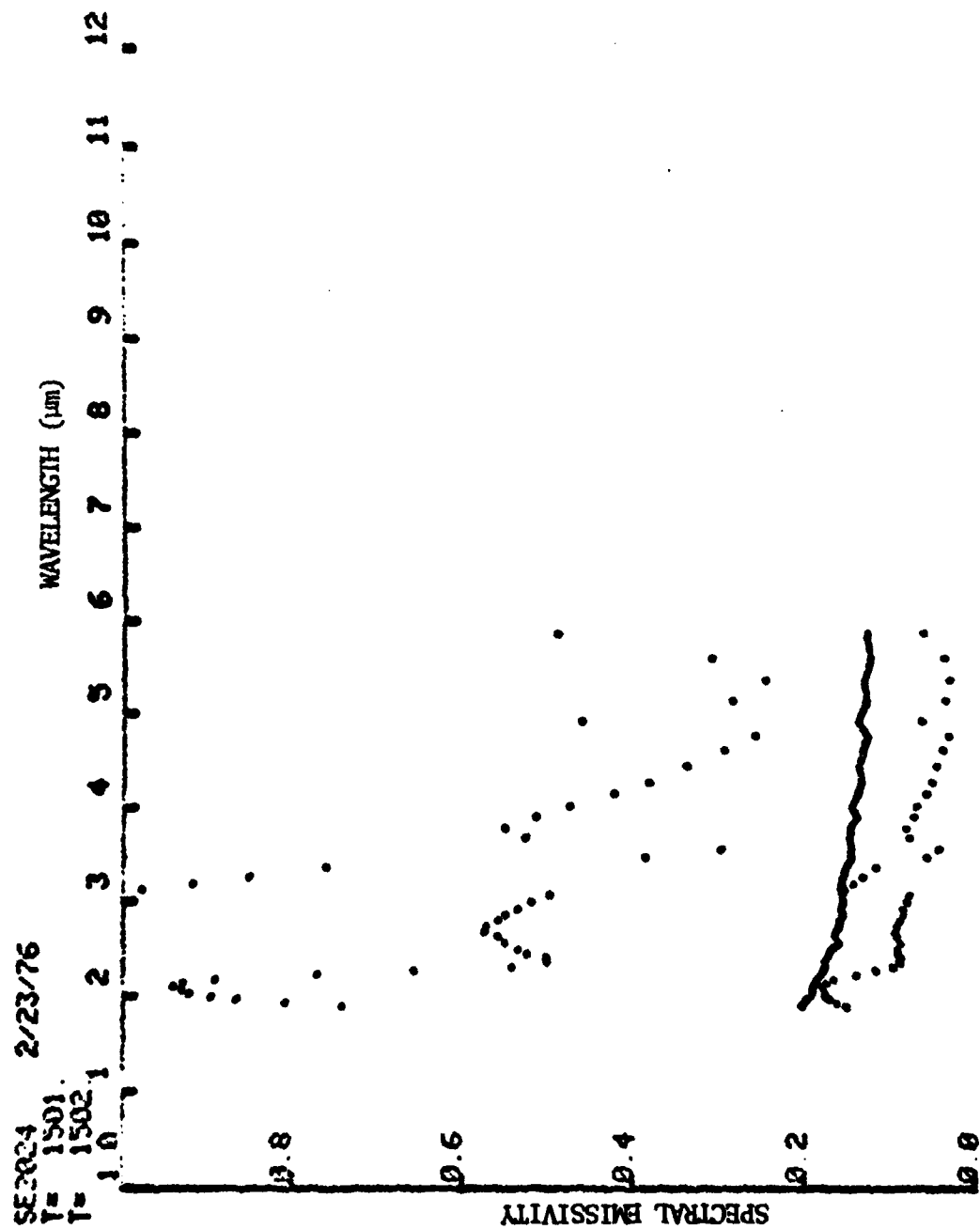
Figure 22. The effect of wavelength interval spacing on the emissivity spectra of tantalum, 2 to 10 μm, (c) repeat of SE2057, Curve (b)

reproducing data accurately with the sodium chloride prism as was found to be the case with the fused quartz prism earlier. It should be noted here also that the results of Figure 22 (SE2024) were taken by the automatic means and Figure 21 (SE2056) by the manual means as mentioned previously.

Comparison of Spectral Emissivity of Tantalum

Figure 23 compares the results of SE2051, Figure 18, with the literature results of Riethof [11]. Since the data by Riethof only covers the spectral range 1 to 4.92µm, the Hagen-Rubens relation was used to extrapolate the emissivity to 12µm [12]. The agreement is generally favorable except for the long wavelength region. This effect is due most likely to stray light typical of single pass prism monochromators and can be minimized by use of proper cutoff filters.

Blackbody Quality Study

In an attempt to evaluate the quality of the sample tube cavity, a series of experiments were performed with the objectives to demonstrate the effect of cavity parameters and to compare observations with the theory of De Vos [2]. The cavity configurations examined are represented in Figure 10. Case I approximates the ideal cavity arrangement wherein the lateral hole is small compared to the diameter of the cavity and the longitudinal extent of the cavity is very large. In Case II and III, the longitudinal dimensions of the cavity are decreased and it is desired to find in what manner the quality is reduced. The configuration of Case III corresponds to the arrangement used for determining the emissivity of nonconducting samples, in cylindrical plug form, which are mounted within the metallic tube.

The pyrometer is focused on one of three targets - upper cavity, sample surface, and lower cavity at three temperatures, namely 1700, 1860 and 2035°K.
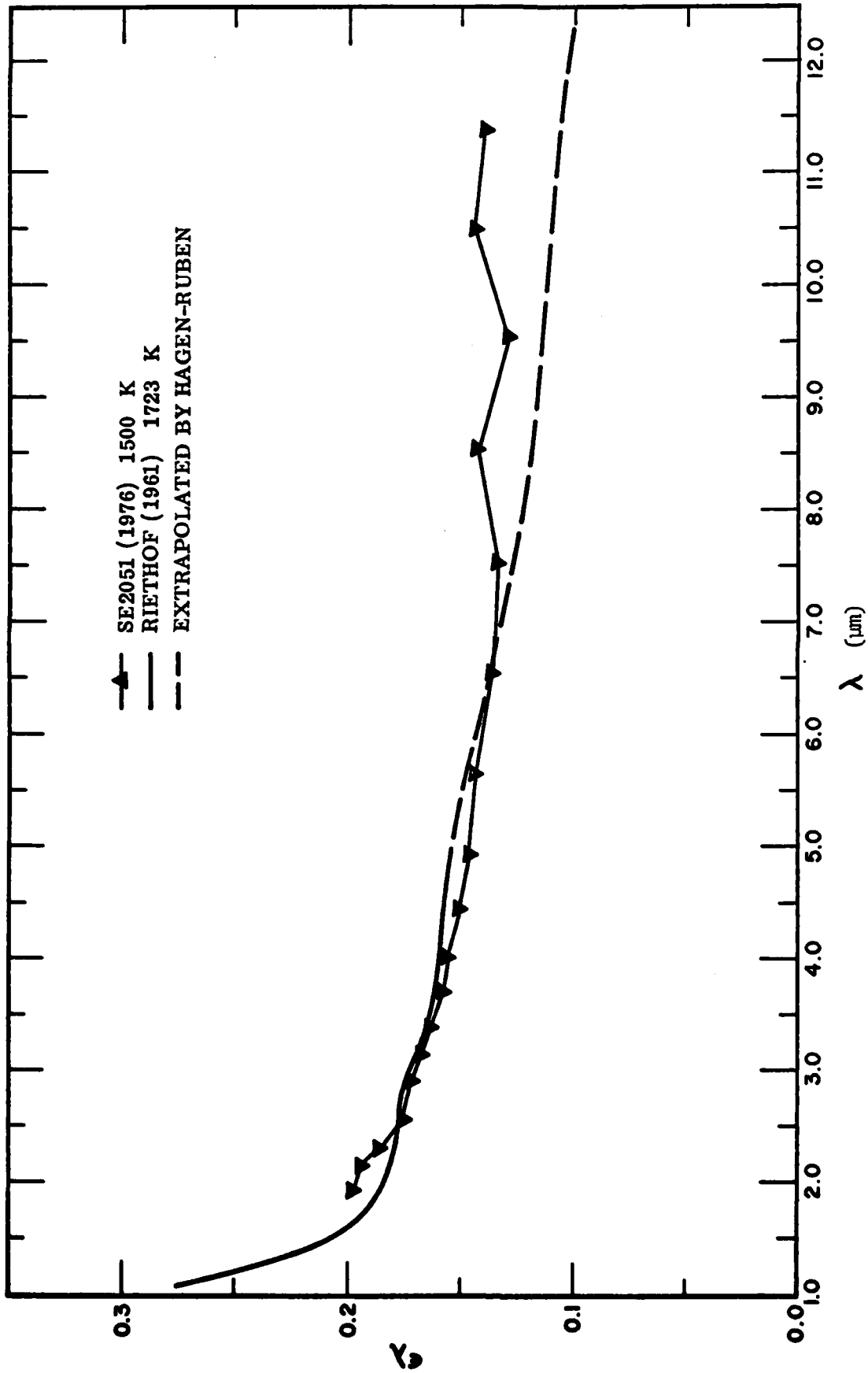
Figure 23. Comparison of spectral emissivity measurements on tantalum with
literature values

44.

Note that in Table 3, for Case I, that the two cavity temperatures $T_{C,L}$ and $T_{C,U}$ are within the stated uncertainty interval of each other and then the sample temperature is taken as the average. The uncertainty limits are determined as the standard deviation based upon 10 to 15 observations taken at 1 second time intervals.

Note that the spectral emissivity of the tantalum tube for the Case I measurements increases slightly (4%) with temperature between 1700 and 1860 K; published data indicate that the temperature coefficient is negative and we should rather have expected a decrease by a like amount. This anomoly could probably be removed by taking greater care to stabilize the surface when changing temperature levels. This effect has little influence on the primary objectives of this study.

Two important features of the results in Table 3 should be noted. First, the emissivity of the tantalum surface showed sufficient variations (6%) that indeed this property could not be used to infer sample temperature. Second, the emissivity of the tantalum showed a systematic decrease--Case III < Case I--within the data set at any one temperature level. This feature cannot be explained on the basis of our procedure or sequence of observations.*

*The observation sequence was: (1) soak at 2000 K for 15 minutes, (2) perform measurements at 1900 K.

Table 3

Temperature Measurements and Emissivity Results

CONFIGURATION	Sample Temperature T, (K)	Sample-Cavity Radiance Temperature and Emissivity			Resistance[3] (Ohm)
		Lower $T_{C,L}$(K)	Sample T_S(K)	Upper $T_{C,U}$(K)	
1	1705.3±0.6	1705.6±0.7 1.000[2]	1604.6±0.8 0.443	1705.0±0.6 1.000[2]	2.43555
2	x	x	x	x	x
3	1704.9[1]	1690.9±0.7 0.898	1600.0±0.4 0.427	x	2.43546
1	1860.0±0.8	1860.9±0.8 1.000[2]	1748.1±1.1 0.464	1859.2±0.6 1.000[2]	2.60502
2	1860.8[1]	1860.5±0.7 0.998	1748.7±1.0 0.466	1859.3±0.7 0.990	2.60728
3	1862.2[1]	1850.9±0.6 0.930	1741.2±0.8 0.435	1721.1±0.9	2.60917
1	2036.7±0.8	2036.4±0.8 1.000[2]	1901.9±0.5 0.463	2037.0±0.7 1.000[2]	2.79875
2	x	x	x	x	x
3	2038.8[1]	2023.9±0.7 0.923	1891.8±0.5 0.430	x	2.80118

Notes: [1] This temperature is computed from resistivity measurements as described in the text.

[2] The emissivity of the cavity is assumed to be unity.

[3] Resistance of test section determined from voltage probe and tube current measurements and used to compute sample temperatures T, for Cases II and III from Case I temperature-resistance data as per Eq. A-4.

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DISCUSSION AND SUMMARY

The high temperature Emissometer has been developed to the state where
spectral emissivity data on both electrically conducting and non-conducting
samples over the spectral region 0.6 to about 7µm can be obtained. At longer
wavelengths, detector sensitivity and signal-to-noise problems severely limit
the accuracy. Improved hardware (a lock-in amplifier and tri-metal detectors)
is being investigated.

The Emissometer has been interfaced to the PRL mini-computer-based digital
data acquisition system (DDAS) and the DDAS controls the experiment, collects
data, computes the spectral emissivity and outputs the results in tabular and
graphical form.

Various factors influencing the performance of the Emissometer have
been evaluated. It was found that the present slit width could be reduced
significantly without affecting the results. It was also found that the over-
all results were not particularly affected by the wavelength increment between
data points, nor by whether the data were obtained during manual or automatic
operation. The run-by-run reproducibility of the data was also found to be
quite good (within the combined uncertainty levels of the individual curves).

The accuracy of the Emissometer was tested using tantalum as a reference
material. Up to 7µm the agreement between the present results and those
believed reliable from the literature are all within five percent and are
generally within two percent. However, above 7µm, the present results are higher
than those obtained by extrapolation using the Hagen-Ruben relation. This may
be due to stray light or to increased signal-to-noise ratios. In either event,
a significant improvement in the operation of the Emissometer at longer wave-
lengths is expected within the next year.

The effect on the emissivity of the cavity at 0.65µm caused by the presence
of the non-conducting sample was investigated. It was found that a sample 14 mm

from the aperture affected the temperature measurements by less than $1^{O}C$ and

affected the emissometer of the "blackbody" hole by 1%. The affect on the

"blackbody" hole 23.8 mm away was 0.2%. However, when the sample was moved in

front of one hole and 7.9 mm from the other, the emissivity of the "blackbody"

decreased to 0.90 - 0.93. The importance of these findings is that the

emittance of the "blackbody" cavity is decreased by the presence of the sample

in front of the other hole and this must be taken into account when measuring

the spectral emittance of non-conductors. Otherwide a 7% error will result.

48.

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49.

APPENDIX A

Description of Computer Program

Overview

At the start of this project (February 1976) there was one primary computer program available for the acquisition of emissivity data. Two additional programs have been generated since that time to provide greater ease in obtaining data. The original program, entitled SE, acquired, analyzed, and displayed the results with a wavelength scale in electron volts, eV. The two new programs are vastly improved versions of the original one, SE, and the wavelength scale is now in micrometers, μm. These programs have been entitled SEM and SEC.

The programs SE and SEM will be discussed in detail along with their respective subprograms and subroutines first because they essentially perform the same operations. Then the third program SEC, which provides greater flexibility for data observations will be discussed. It should be noted that program SE is now obsolete. This is because SEM and SEC have overcome many of the problems or limitations that existed with the original program, SE. Also SEM and SEC include more override types of commands that are useful to the operator during a data run.

Programs SE and SEM

The actual acquisition of data takes place in the following order:
(a) The initial parameters are keyed into the computer at the start of programs SE and SEM. These parameters include:

    (a) the desired wavelength interval between data points in units of 0.01 eV for SR or 0.01 μm for SEM;

    (b) the pyrometer range (dependent on the temperature range of the run) 1, 2, or 3;

(c)   the delay time allowed for the detection system to stabilize in 0.1 seconds;

(d)   the initial energy position for the first data point in units of 0.01 eV or 0.01 μm; and

(e)   a specification as to which prism is being used in the spectrometer, 1 for sodium chloride or 3 for fused quartz;

(f)   chains to program TT or TTM.

(B)   Under program TT and TTM the following occurs:

(a)   the program opens a data file in the computer;

(b)   determines the temperature of the sample holder tube by using the stored pyrometer calibration information;

(c)   obtains a radiometric zero reading (graphite block to the right of and just behind the sample holder) with detection system in the spectral mode;

(d)   mirror 1, denoted M-1 on Figures 6, 7 and 9, is cycled for alignment;

(e)   the spectrometer wavelength drive motor is engaged and run till the predesignated initial energy (eV) position, see step (A,d), is found and then stops the motor; and finally

(f)   subroutine TD or TDM is called to take data.

(C)   Under subroutine TD:

(a)   the drive motor on the spectrometer is engaged and run till the next energy (eV or μm) position is achieved which is designated by the energy differential between data points of item (A,a) (Note that this command is ignored for the initial data point).;

(b)   mirror M-1 is set to take the blackbody reading (lower hole of the tantalum tube sample holder);

(c)   the computer waits for the detection system to stabilize as per step (A,c);

(d)   determination of whether a scale change is needed or not is made and if so returns the program to step (C,b) after the lock-in amplifier gain setting has been readjusted manually;

(e)   data is taken and temporarily stored by the computer;

(f)   mirror M-1 is now switched to take the sample data. Mirror M-1 can be positioned to focus on the upper hole in the metallic tube, where a nonconducting sample material could be or on some intermediate target between the two holes on the metal tube. In the latter position, the sample target would be the metallic tube itself. For a 100% level test M-1 would be adjusted so both holes can be viewed;

(g)   again, after the detection system has stabilized, the sample data is taken and temporarily stored;

(h)   all the data at this point, both blackbody and sample, are corrected to the zero temperature signal of step (B,c) and permanently recorded on file;

(i)   ε is then determined in the ratio of the corrected sample over the corrected blackbody and is plotted on the video screen of the computer

(j)   subroutine TD_ is repeated until the signal-to-noise from the spectrometer is weakened or until the wavelength spectrometer has reached approximately 0.1 eV for SE or 12 μm for SEM; and

(k)   then subroutine TD_ returns back to the respective TT_ subprogram.

(D)   Under subroutine TDM the following steps from subroutine TD are followed:

(a)   step (C,a);

(b)   M-1 is refocused on the zero temperature reference and a new zero reading is obtained as in step (B,c);

(c) step (C,b) to (C,i) are completed;

(d) the pyrometer range is again keyed into the computer;

(e) a new temperature reading is determined from the pyrometer observation

(f) the computer prints out on the line printer the data of that particular data point for immediate evaluation. Data includes: λ, H, S, ϵ, temperature and Z; and

(g) finally steps (C,j) and (C,k) are completed.

(E) Finally under program TT or TTM

(a) a final or end-of-run temperature is determined from the pyrometer observation;

(b) the data files are closed and the data acquisition run is complete.

Program SEC

The third program called SEC, is used when the operator wishes to change the temperature and/or wavelength during the data run. SEC is similar to SEM in that it operates with a wavelength scale in μm. The only major difference lies in the fact that the differential energy between data points and the initial energy position, see items (A,a) and (A,d) of programs SE and SEM, are meaningless and are therefore deleted. With these exceptions noted in SEC, we will begin the discussion of program TTC.

TTC is also similar to its counterparts TTM and TT with the one following exception: Item (B,e) from the discussion of TT and TTM has been deleted. Since one constant wavelength may be desired, the drive motor on the prism spectrometer need not be used.

Under subroutine TDC the following steps from subrountine TD are followed:

(a) M-1 is focused on the radiometric zero and a reading is obtained;

(b) steps (C,b) to (C,i) are followed;

(c) the pyrometer range is again keyed into the computer;

(d) a new temperature reading is determined with the precalibrated
     pyrometer;

(e) the computer prints out on the line printer the data for that
     point for immediate evaluation. Data includes λ, H, S, ϵ, tempera-
     ture, and Z.

TDC repeats the above five steps for as many data points at various tempera-
ture and wavelengths as may be desired. Then under command of the operator,
the program returns to TTC and proceeds to close the data file.

Operating Options

As can be seen from the previous section there exist certain options
available to the operator while data is being taken. To summarize them
categorically under program SE, the operator could, during a data run,

(1) halt the data run at any time;

(2) adjust the number of data points being averaged to determine a
     singular data point to be recorded; and

(3) take repeat readings at a particular wavelength.

Under SEM the operator may during a data run

(1) again halt the data run at any time;

(2) adjust the number of data points being averaged to determine a
     singular data point to be recorded;

(3) take repeat readings at a particular wavelength;

(4) readjust the lock-in amplifier to a better signal level without
     restarting a data run;

(5) know the value of a data point being stored in memory at that
     same instant of storage.

Finally under SEC, each of the options, 1-5, of SEM plus the following
are available

(6) change the temperature while at one wavelength, this usually
    necessitates readjustment of the lock-in amplifier-gain setting
    which may be done without interference to taking data;

(7) change the temperature and the wavelength and again readjust the
    lock-in amplifier gain setting;

(8) or finally change the wavelength to some predetermined one and
    maintain the same temperature. Likewise readjustment of the lock-in
    amplifier gain setting is again available; and

(9) any combination of the above items, 6-8, under this section for
    SEC.

Comparison Programs

In completion of this section on measurement procedures with computer
aids, three more computer programs which are used will now be presented. The
first is entitled SEL. This program will take two previous data files which
were previously stored in the computer and display them on a video screen
to be either photo copied and/or merely re-examined. The second program
is called OFR. This program also requires a previously stored data file.
This program has been designed to determine if an old data file is in wave-
length units of eV or μm and then plot the results or data on the display
screen again for the purpose of photocopying. Note that this program will
automatically, before the data is plotted, convert any old data which might
be recorded in eV units to μm units. The third program is entitled, CF,
for Change Files, and is incorporated into OFR. These last two programs,
OFR and CF, were mainly conceived to eliminate the need to convert old data
and plotted graphs from eV units to μm units. Copies of SE, SEM, SEC, TT,
TTM, TTC, TD, TDM, TDC, SEL, OFR and CF subroutines are available but not
included in this report.

APPENDIX B

List of Current Computer Programs

Number	Designation	Title/Function
1	CF	Change File/changes files to micrometer form
2	OFR	Old File Replotted/replots an old file
3	SE	Spectral Emissivity/setup to take spectral emissivity varying wavelength automatically
4	SEM	Spectral Emissivity Micrometer form/program SE using micrometers
5	SEC	Spectral Emissivity Constant temperature or wavelength/program SEM and not varying wavelength
6	SEL	SE Extension/plots two previous SE files
7	TD	Take Data/actually takes the data for SE
8	TDC	Take Data Constant temperature or wavelength/like TD but for SEC
9	TDM	Take Data Micrometer form/like TD but for SEM
10	TT	Take Temperature/actually takes temperature reading for SE
11	TTC	Take Temperature Constant temperature or wavelength/like TT but for SEC
12	TTM	Take Temperature Micrometer form/like TT but for SEM